Microstructure evolution in irons and steels: a tribute to David V. Edmonds

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The contributions of David Vernon Edmonds to the study of microstructural evolution over the past four decades are reviewed, in a tribute to mark his recent retirement from the Chair of Metallurgy and Materials at the University of Leeds. The scientific discoveries in the Edmonds group are highlighted in the context of microalloyed, ferritic, pearlitic, bainitic and martensitic steels, as well as cementite and cast iron, and in a variety of non-ferrous fields including high-density tungsten and uranium alloys, titanium alloys and twin-roll thin-strip casting.

Keywords: Microstructure evolution, Bainite, Interphase, Ferrite, Cementite, Graphite

Introduction

In celebrating the career of Professor David Edmonds, we recognise the central theme of his work on solid-state phase transformations and microstructural evolution involving the application of a variety of metallographic techniques, where he and his research group have made important contributions over four decades. David Edmonds pursued his BSc and PhD in physical metallurgy at the University of Birmingham. His doctoral studies on yield phenomena in zirconium and hydride embrittlement of titanium were with Jim Beevers, a former student of Professor Robert Honeycombe at Sheffield. This connection led David to join Honeycombe's group at Cambridge, working on the interphase precipitation of particles at moving ferrite/austenite interfaces in alloy steels as the initial stage of what were 11 exceptional years at Cambridge (1968–79). In 1979 David joined the Department of Metallurgy and Science of Materials at Oxford as a lecturer in Industrial Metallurgy. He was recruited to Oxford in part to help create the MEM (Metallurgy, Economics and Management) programme, designed to nurture captains of engineering industry. This option involved an extra year of study, including a management-training internship in industry, and remains today with a more fashionable title. In 1993, David was elevated to the Chair of Metallurgy and Materials, formerly held by Jack Nutting, at the University of Leeds. This was a period of great change in the world of materials and David served two terms as the Head of Department over the periods 1995–98 and 2001–04, during which the activity metamorphosed into what is now the Institute for Materials Research, in the School of Process, Environmental and Materials Engineering at Leeds.

David V. Edmonds

David's endeavours in science have been recognised most prominently by his election to the Royal Academy of Engineering and the Fellowship of ASM International. But there are many other marks of respect for his work, including the Vanadium Award from the Institute of Materials, Minerals and Mining in both 1993 and 2002. David has always given teaching a high priority. The Oxford 'tutorial system' that he participated in is inherently hard work but enjoyable. Over the years, he has succeeded in explaining an impressive range of metallurgical subjects to students, including solid state phase transformations, deformation, fracture, fatigue, creep, ferrous and other alloys, introductory materials science and engineering, along with a variety of processing-related aspects such as casting, working, welding, machining, surface finishing, heat treating.

The authors of this tribute were privileged to have their doctoral research supervised by David during his years at Cambridge (HKDHB, 1976–79) and Oxford (JGS, 1980–83). In the sections that follow, we highlight some of the key developments attributed to the Edmonds research group, focusing on the most stable element in the universe, iron.

Microalloying

Seminal work using transmission electron microscopy (TEM), begun at Sheffield and continued at Cambridge by Honeycombe and co-workers,[1,2] had revealed two distinct forms of alloy carbide or carbonitride precipitation in ferrite. It seemed that tiny particles precipitated in arrays of parallel sheets, because they formed at austenite/ferrite transformation interfaces. This interphase

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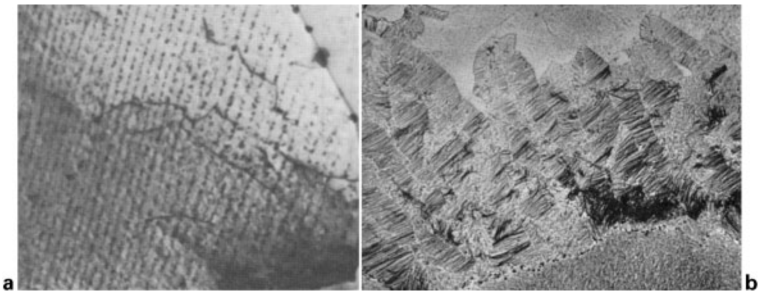
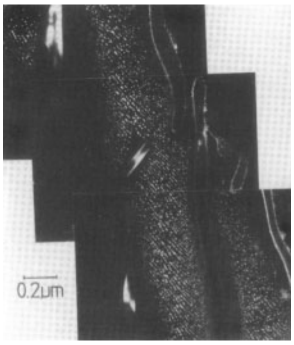


1 **Electron micrographs of *a* interphase[3] (×35 000) and *b* fibrous precipitation[9] (×4000) of alloy carbides in pro-eutectoid ferrite**

precipitation and associated microstructures consisting of arrays of long fibres, was visually striking and remarkable in its organisation (Fig. 1). The arrays were found in the then emerging microalloyed steels and in heat-resistant power plant steels, and David was engaged by their effects on strength,[3] creep,[4,5] fracture[6] and fatigue.[7,8] His enthusiasm for high resolution microanalytical techniques was boosted by these newly-discovered forms of precipitation, especially the fibrous form.[9,10] He pioneered the application of photoemission electron microscopy to look at the austenite as it decomposes,[11–13] thus revealing for the first time, direct observations of the propagation of the transformation interface by a step mechanism, in a manner consistent with interphase precipitation.[14] This work led to a more general interest in austenite formation and decomposition, particularly in ways which circumvent the loss of parent austenite in partially transformed samples by martensitic transformation, an example being use of thin retained austenite films in the martensite[15] to relate the three-phase crystallography at the interface between parent austenite and product ferrite and 'interphase-precipitated' carbide and explain therefore the selection of particular crystallographic variants.[16]

Another way to preserve austenite was with a high-Mn alloy such as Hadfield steel, which is typically austenitic at room temperature but can be in a state where cementite, ferrite and austenite are in three-phase equilibrium. This permitted the direct observation of the austenite/ferrite interface. In a microalloyed version, it helped to demonstrate the possibility of alloy carbides in pearlitic ferrite. This is of use in steels for forgings, rod, wire and rails,[17–19] the precipitation occurring via an 'interphase' mechanism (Fig. 2).[20,21] Meanwhile, it became evident that microalloying elements can be exploited for applications other than grain refinement and precipitation strengthening. It was shown that minor amounts of V (and/or Si) can modify the initiation of austenite decomposition at the grain boundaries in high carbon rod steels (Fig. 3).[22] This mechanism was used to eliminate cementite films which embrittle grain boundaries in hypereutectoid steels, enabling the development of pearlitic wires stronger than 2350 MPa at practical diameters.



2 **Interphase precipitation in pearlitic ferrite (dark-field electron micrograph)[16]**

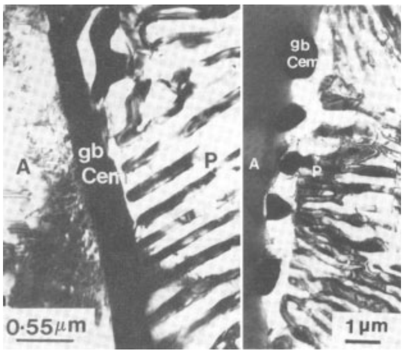
Pearlite

In a programme to study stress relaxation in spring steels, cold-drawn after patenting, atom-probe field ion microscopy was used to obtain one of the first direct observations of interstitial atom decoration of dislocation substructure (Fig. 4).[23,24] The dislocation substructure changed with low temperature heat treatment whereas the interstitial atmosphere was virtually unaffected.

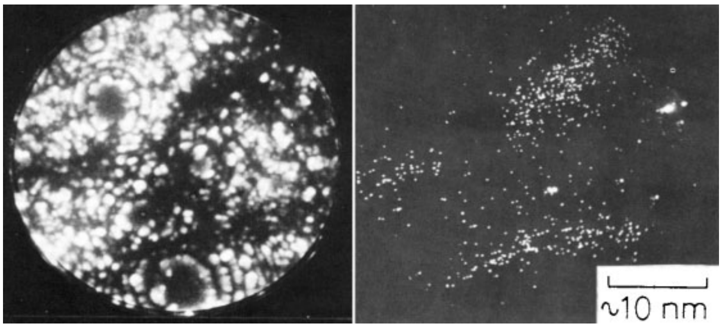
Constant growth rate (forced velocity) experiments, instead of isothermal heat treatments, were also conducted on eutectoid and off-eutectoid steels[25–27] utilising a steep temperature gradient to move a single transformation interface through the material at a controlled rate. These studies helped determine the diffusional transformation mechanism from the measured velocity/interlamellar spacing relationship, the extent of cooperative growth and the ability to align the lamellar microstructure; experiments were also extended to Cu–In alloys.

Bainite

The similarity between variant selection of interphase precipitation and that of cementite precipitation in lower



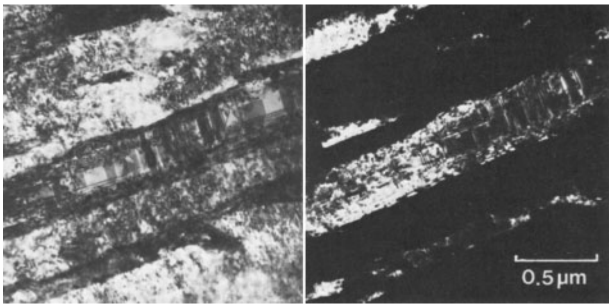
3 **Electron micrographs illustrating modification of grain boundary cementite precipitation in hypereutectoid steel (left) by vanadium addition (right)[22]**



4 Imaging atom probe micrographs indicating carbon segregation to dislocation substructure in cold-drawn eutectoid steel wire[24]

bainite, and also the important experience of examining the ledged austenite/ferrite interfaces in reconstructive transformations, led to studies of the bainite transformation in the Edmonds group. At that time the bainite transformation appeared little understood as compared with, say, the martensite or pearlite transformations, which was partly responsible for less interest in the development of commercial bainitic steels. Thus began an interesting period exploring the bainite transformation mechanism, particularly with respect to its displacive or martensitic characteristics,[28,29] and this work was followed by efforts to begin to develop a microstructural route to tough, high strength steels based upon bainite.[30–36] The experimental approach followed the seminal work of Hehemann and co-workers,[37–39] using Si alloying to suppress cementite formation so that austenite decomposition to bainitic ferrite could be examined unhindered by the secondary reaction. The displacive mechanism remained controversial at the time[40–42] and to this day, but the work initiated a fairly wide and lengthy debate which at a minimum corrected the previous neglect of bainite as a useful microstructure and must in some way have contributed towards the subsequent emergence of transformation-induced placticity-assisted steels[43–45] which are industrially important, and low-temperature transformed 'nanobainite' structures that have emerged more recently.[46,47]

The new microstructural route to improved performance that has emerged is based upon carbide-free bainitic ferrite, brought about by the Si alloying addition (or Al additions in more recent studies). However, another microstructural feature is equally important, which is the presence of interlath retained austenite, stabilised by the unprecipitated carbon[48] (Fig. 5). These two microstructural features provide the potential for mechanical property improvements: high strength, toughness, fatigue resistance and, because austenite might act as a sink for hydrogen as well as carbon, hydrogen embrittlement resistance. These concepts also hold for the newer quenched and partitioned (QP) steels,[49–51] which essentially exchange martensitic ferrite for bainitic ferrite, but with more control over the carbon concentration of the retained austenite phase.



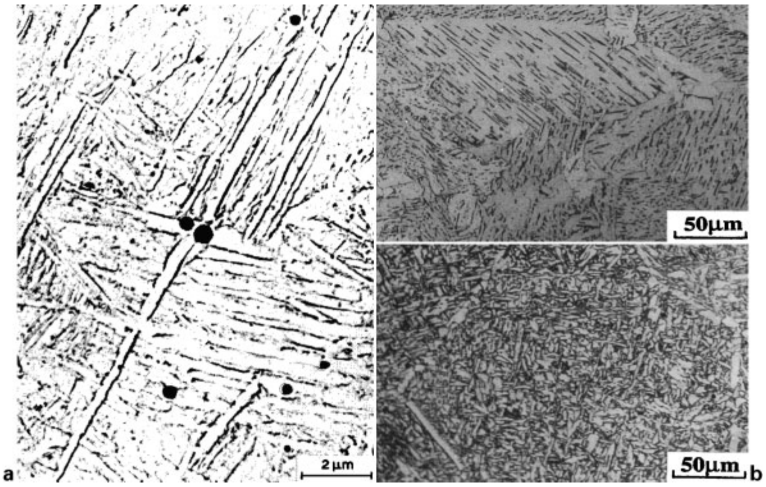
5 Electron micrographs of carbide-free bainite in high-Si steel: interlath retained austenite is revealed by internal stacking faults and dark-field imaging[48]

Acicular ferrite

With his interest in bainite and martensite, it was perhaps not unnatural that David turned his attention to a study of the so-called 'acicular ferrite' microstructure. This microstructure became a hot topic during the 1970s in the development of tough weld metals when a general consensus was emerging that intragranular nucleation at weld metal inclusions was responsible for the intragranular nucleation of acicular ferrite.[52] The Edmonds group contributed persuasive metallographic evidence for inclusion-assisted nucleation and sympathetic nucleation (Fig. 6*a*) along with the dependence upon inclusion content, austenite grain size and Mn concentration.[53] In the 1990s David returned to the topic, this time in order to explore the potential for developing this strong tough microstructure in wrought steels, and also to investigate the so-called 'vanadium effect' whereby this element seemed to contribute towards an intragranularly-nucleated acicular ferrite structure.[54,55] Good metallographic evidence of the 'vanadium effect' was found (Fig. 6*b*), but in this instance using high-purity laboratory-produced alloys, with an absence of any significant inclusion-assisted nucleation, and no visible vanadium carbonitride precipitation. High resolution techniques of scanning transmission electron microscopy coupled with energy dispersive X-ray analysis and secondary ion mass spectrometry indicated vanadium segregation within the microstructure, which may suggest that solute vanadium contributes to intragranular acicular ferrite formation via its interfacial or ferrite stabilising effects.

Austempered ductile iron

The bainitic structures studied in steels alloyed with Si, led to an interest in austempered ductile cast irons, which, because of their similarly high Si content and austempering treatment possess equivalent carbide-free bainitic microstructures containing retained austenite. TEM studies by the Edmonds group characterised the bainite matrix which, although confirmed to be similar to that previously studied in steels, can contain



6 *a* electron metallographic evidence for inclusion-assisted intragranular nucleation of acicular ferrite in submerged arc weld metal[53] and *b* light micrographs illustrating influence of vanadium addition (bottom) promoting acicular ferrite microstructure in 0·1 wt-%C steel[55]

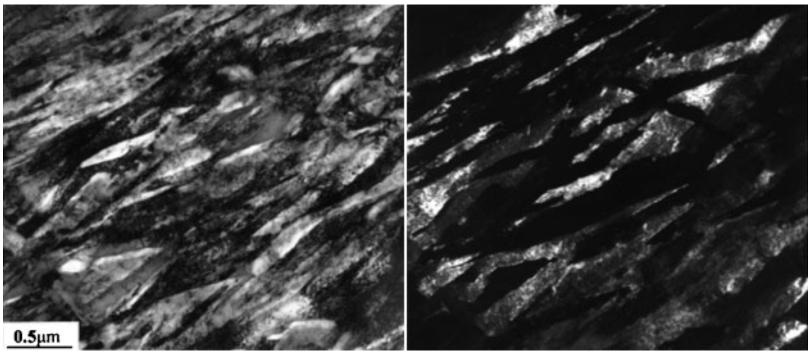
segregation of the main alloying elements that influence final microstructure; Si, Ni and Cu graphitisers in the eutectic cell, and Mn, Mo carbide promoters in intercellular regions. The concentration gradients across the microstructure probably account for a number of carbides which were identified; cementite, epsilon, eta, kappa, and faulted triclinic carbide previously identified in high-Si steels.[56,57] Later studies concentrated on the effects of substituting aluminium for silicon, which resulted in a similar matrix microstructure of carbide-free bainite with interlath retained austenite but also, importantly, enhanced oxidation resistance.[58]

Quenched and partitioned steels

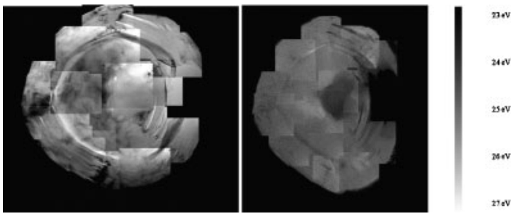
Quenching and partitioning is a new concept for the heat treatment of martensite, somewhat different than customary quenching and tempering. The process involves quenching to below the martensite-start temperature and directly annealing, either at, or above, the initial quench temperature. If competing reactions, principally carbide precipitation, are suppressed by appropriate alloying, the carbon partitions from the supersaturated martensite phase to the untransformed austenite phase, thereby increasing its stability and retention upon cooling to room temperature (Fig. 7). A key to successful heat treatment is prevention of carbide precipitation and so recent metallographic studies[59–62] have focused upon the conditions controlling precipitation of cementite and the transitional epsilon carbide, utilising mainly high resolution TEM but also the position-sensitive atom probe. David's group at Leeds has been instrumental in the characterisation of QP microstructures, and particularly in helping to understand the transition carbide precipitation behaviour in both low and medium carbon alloys subjected to QP processing. The importance of transition carbide solubility/stability has been critical in understanding how large quantities of austenite can be stabilised by carbon partitioning in some processing scenarios, while fine transition carbides are more common in others.

Graphitisation

Solid-state nucleation and growth of graphite nodules in steels emerged from Edmonds' sustained interest in the effects of Si alloying combined with the more recent idea to explore the use of graphite as a route to improved machinability of steels, rather than additions of Pb, S, Te, Bi or P, which may impair cold forgeability, or create toxicity and environmental problems rendering the steel expensive and difficult to recycle. The equilibrium form of carbon in the Fe–C system is graphite, but kinetic considerations ensure that under normal heat treatments iron carbide (cementite) forms first. Long term annealing can replace cementite by graphite but is not a commercially-viable industrial



7 Bright- and dark-field electron micrographs of typical interwoven martensitic ferrite and retained austenite of QP microstructure[60]



8 **Bright-field TEM montage image and plasmon ratio map indicating less graphitic nature of core compared with outer layers of graphite nodule about 2 μm in diameter[66]**

process. However, by increasing the concentration of graphitising elements, particularly Si, and reducing carbide forming elements, graphitising times were significantly reduced to just a few hours.[63–65] Significantly, the experimental results, obtained by high resolution TEM and utilising a quantitative method of mapping plasmon energy loss shifts within graphitising carbon systems using electron spectroscopic imaging (Fig. 8), indicated that the refined dispersion of small graphite nodules nucleated from temper particles associated with cementite during the early stages of the graphitising anneal.[66–69] Consistent with this hypothesis is the observation that graphitising kinetics are influenced by the starting microstructure, either quenched martensite, austempered (high-Si) bainite or pearlite.

Proeutectoid Widmanstätten cementite

The high-Mn Hadfield steels used to study features of the austenite/ferrite decomposition interface and interphase precipitation in pearlitic ferrite exhibit hypereutectoid behaviour and presented an opportunity also to examine cementite precipitation in austenite[70] as well as the unexpected precipitation of elemental copper in cementite. Widmanstätten precipitation of proeutectoid cementite in hypereutectoid steels has not been studied extensively. However, the high-Mn alloy allowed a detailed study of its morphology, interface structure and orientation relationships to the parent austenite phase, and revealed a completely new orientation relationship,[71] within 3° of $(100)_C \| (112)_A$, $(010)_C \| (02\bar{1})_A$ and $(001)_C \| (\bar{5}12)_A$. The morphology of the plates possessing this new orientation relationship is distinctly different from that of plates with the more familiar Pitsch orientation relationship[72] (Fig. 9). These different Widmanstätten plates were subsequently shown also to occur in plain carbon steel, using retained austenite films in the martensite matrix to identify and confirm the new orientation relationship.

Elemental copper was found to precipitate in proeutectoid ferrite and pearlitic ferrite similarly to alloy carbides, and therefore by an assumed interphase precipitation mechanism. Unexpectedly, however, copper was also observed to precipitate in the high-Mn hypereutectoid alloys in proeutectoid or pearlitic cementite[73–77] (Fig. 10). Besides opening up an interesting topic for more fundamental study (e.g. the copper can act as a marker of the progress of the transformation), this discovery may give the opportunity to improve the crack resistance of the cementite phase, and hence the fracture toughness of plain carbon steels and cast irons.

Anomalous or irregular microstructures

Ultra-low carbon steels

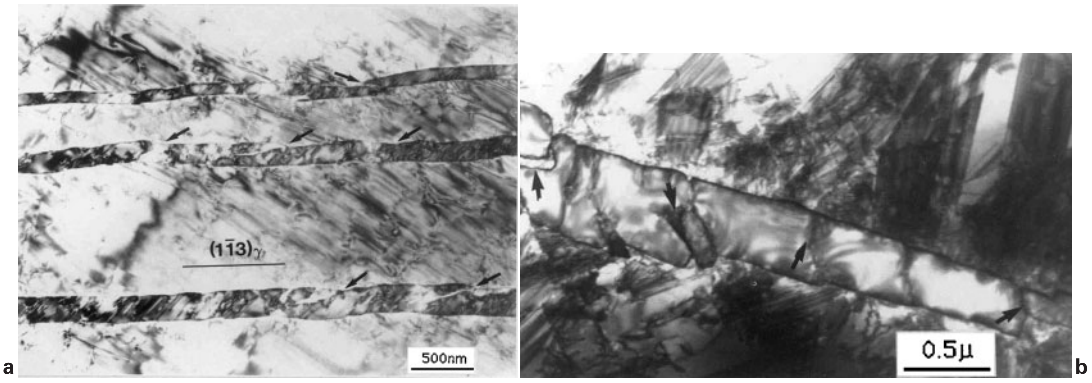
Developments in modern steelmaking techniques have allowed the control of carbon concentrations to low levels and the ferritic transformation microstructures at these carbon levels are sometimes not well understood. A systematic metallographic study of ferrite in ultra-low carbon iron alloys helps in classifying the microstructural forms and identifying potential mechanisms of formation as the steels tend towards pure iron and massive transformation.[78]

Abnormal ferrite

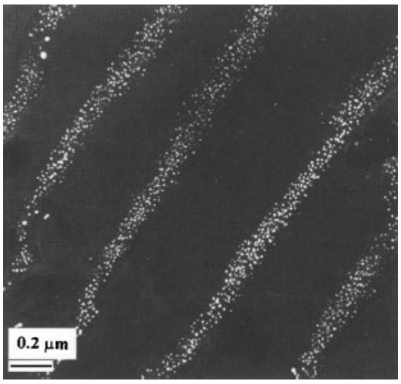
A less understood but well-documented phenomenon in high-carbon steels is that ferrite can form in hypereutectoid compositions decomposed at small undercoolings beneath the eutectoid temperature. Well-known to occur in carburised steels it has been termed 'abnormal' ferrite. Observations have shown that this ferrite forms on the proeutectoid cementite which can continue to grow, thus suppressing the 'cooperative growth' conditions required for pearlite, and a model for the carbon redistribution required was developed[79] for this structure.

Cr steels

An unusual acicular morphology of ferrite/carbide aggregate in Cr-containing steels which forms in a temperature range between classical pearlite and classical bainite structures has been examined and attributed to a transformation mechanism analogous to that of inverse bainite, where ferrite coats proeutectoid cementite, but in this case, a chromium carbide phase.[80]



9 **Electron micrographs of proeutectoid Widmanstätten cementite plates in high-Mn steel related to parent austenite by *a* Pitsch orientation relationship and *b* Farooque/Edmonds orientation relationship[72]**



10 **Dark-field electron micrograph illustrating copper precipitation within thinner lamellae of pearlitic cementite[77]**

Steel corrosion

This work was conceived to investigate the resistance of modern microalloyed steels to CO_2 attack, the most prevalent form of corrosion encountered in oil and gas production and transmission, where traditional carbon and low alloy steels have poor resistance, forcing alternative expensive solutions. The programme paid special attention both to steel alloying and to processing and heat treatment, and tested a number of microalloyed steels in simulated field environments. A fivefold improvement over X70 in CO_2 corrosion resistance at less than 1·5 times cost was demonstrated (Fig. 11); in particular, vanadium was identified as a useful microalloying addition in this environment.[81]

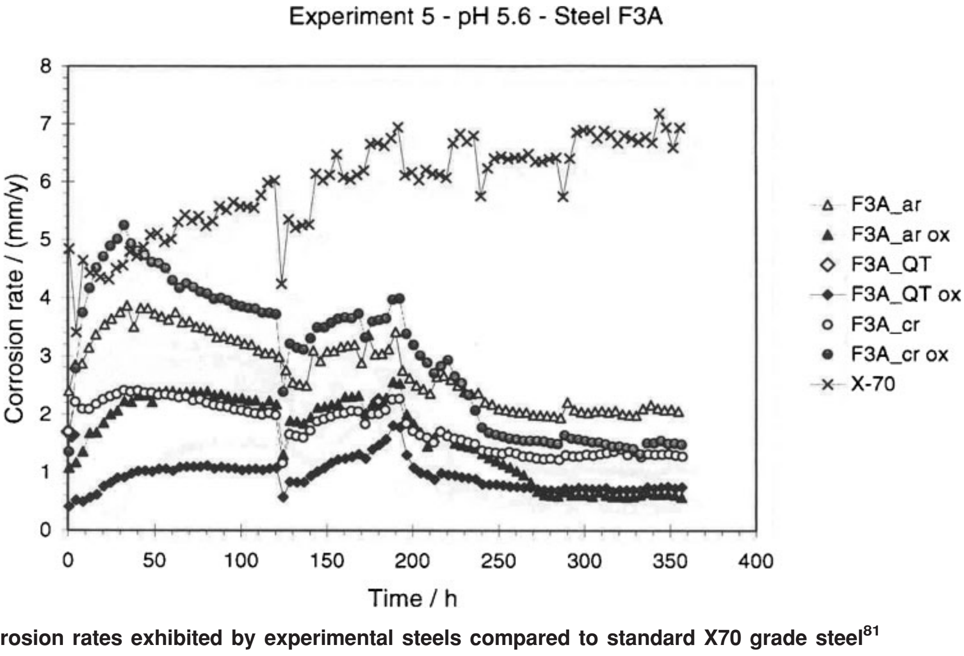
Additional contributions by the Edmonds group have been made to steel research in such areas as dual phase,[82] martensitic[83] and austenitic steels.[84]

Non-ferrous alloys

David Edmonds' research in steels for defence applications led to interest in non-ferrous alloys with high density for kinetic energy armour penetration. Impurity segregation to microstructural interfaces in liquid phase sintered tungsten heavy alloys, and precipitation of carbide and intermetallic phases, both of which reduce the mechanical properties, particularly toughness, were identified in W–Ni–Cu and W–Ni–Fe by TEM and early Auger spectroscopy.[85–89] Depleted uranium alloys provide an alternative high-density system, and three binary systems of uranium were studied: U–Ti, U–Mo and U–Nb. Techniques using TEM and atom-probe field ion microscopy characterised the martensite transformations,[90,91] aging reactions and isothermal decomposition structures in these alloys,[92,93] along with deformation microstructures.[94] The emerging position-sensitive atom probe technique provided persuasive evidence for spinodal decomposition in U–6Nb, although still under discussion.[95] Also related to ballistic applications, complex deformation structures developed in FCC copper and BCC iron over the strain-rate range 10^3 to $>10^{-5}$ s^{-1}, achieved by Hopkinson bar tests and explosively forming projectiles, were examined by TEM.[96] Ultra-high strain rate deformation is not well understood, although important in modelling and understanding ordnance applications, explosions, high-speed impacts and earthquakes.

The use of hydrogen as a temporary alloying element during fabrication of Ti-matrix composites and heat treatment of Ti_3Al based aluminides was explored. Hydrogen is a beta-stabiliser and prehydrogenation and post-dehydrogenation treatment lowers the phase transformation temperatures, and hence fabrication and heat treatment temperatures, to give less interface degradation between fibre and matrix in Ti-based metal-matrix composites[97] as well as refined microstructure in the titanium aluminides.[98,99]

An important departure from microstructural investigations during David's career was to join with John Hunt at Oxford in a joint industry-university initiative to upgrade the performance of the twin-roll thin-strip casting process.[100–103] Features limiting high-speed thin-strip casting were investigated on laboratory-scale casters, and aluminium alloys were cast at thinner gauges and higher productivities than were customary with commercial machines, enabling the revolutionary design and installation of twin-roll



11 **Lower corrosion rates exhibited by experimental steels compared to standard X70 grade steel[81]**

casters (Davy Fastcast) in two European plants. These facilities cast 10 ton coils at speeds up to 10 m $\min^{-1}$ and thicknesses down to 1 mm in AA 8xxx series alloys.

Acknowledgements

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