

# The recent development of steels with carbide-free acicular microstructures containing retained austenite

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*After a lengthy period of improvement in carbon steel ferrite/pearlite microstructures by microalloying and controlled processing, some attention has focused back more recently upon acicular forms of microstructure. In particular, an interesting advance in this area has been the development, primarily by alloying with Si, of so-called 'carbide-free' bainitic steels, where carbon-stabilized retained austenite is substituted for cementite.*

*Transformation induced plasticity (TRIP) sheet steel with enhanced properties, principally targeted for automotive use, and future potential 'nanobainite' steel, are two noteworthy examples. Even more recently, this concept of developing steel microstructures containing carbon-enriched retained austenite has been extended further by introducing novel heat treatment procedures to replace bainite with martensite. This non-equilibrium 'quenching and partitioning' process route, as it is known, offers possibilities of immediate advantage: enhanced strength from a martensitic structure protected from the more damaging effects of carbon, along with the promise of new properties from the retained austenite phase which could potentially contain a very high controlled concentration of carbon.*

## KEYWORDS:

Quenched and Partitioned (Q&P) Steels; Retained Austenite; Bainite; Martensite; TRIP Steels

## INTRODUCTION

One of the most important steel developments in the last part of the 20th century involved microalloying and special processing of constructional and automotive steels through grain refinement and carbide precipitation [e.g. 1-3]. These microalloyed steels were based upon ferritic microstructures, having a largely equiaxed morphology, with a trend towards reduced carbon levels to enhance properties, improve weldability, and facilitate continuous casting. Even in conventional quenched and tempered martensitic steels it is necessary to control the effects of carbon, which, although imparting strength can also lead to a variety of complications e.g. quench cracking or temper embrittlement. However, carbon is a relatively cheap alloying element and responsible for a variety of microstructures and properties it confers upon iron through heat treatment, which has enabled iron and steel to develop as dominant engineering materials. The use of carbon in a different novel sense opens up possibilities for developing new forms of steels and heat treatment. This is the case with the recent emergence of steels with acicular microstructural constituents based upon carbide-free bainitic or martensitic microstructures containing retained austenite. In consequence, for this paper, ultra-low carbon bainitic steels are

not considered because carbon is not a key alloying ingredient. The contrasting philosophy in acicular steels with retained austenite is to prevent carbide formation (hence 'carbide-free'), thereby improving mechanical properties, not by reducing carbon, but by moving carbon from the acicular ferritic phase (where, in a general sense, it could be damaging to certain properties) to the retained austenite second phase. Firstly, therefore, the austenite phase acts as a potential carbon sink to protect the ferrite from any problems such as those mentioned above. Secondly, the carbon then chemically stabilises the austenite against further decomposition. (This also contrasts with conventional martensite tempering, where in order to stabilise the final microstructure, further decomposition of untransformed austenite is encouraged.) In consequence, the refined acicular microstructure can promote strength, whilst the presence of the ductile fcc austenite second phase can potentially buffer crack propagation to enhance toughness, or alternatively, undergo mechanically-assisted transformation to increase work hardening and uniform elongation, and hence formability.

## CARBIDE-FREE BAINITIC STEELS

It is generally accepted that austenite can decompose to acicular bainitic ferrite with or without carbide formation [4]. Such carbide-free bainite generally consists of bainitic ferrite interwoven with retained austenite (Fig. 1 [5]). The austenite phase is retained due to chemical stabilization from increased carbon concentration, resulting from suppression, usually by alloying with Si, of precipitation of the cementite form of iron carbide. Although there may be earlier beginnings, it might be argued that the main interest in the carbide-free form of bainite in Si-containing steels, which led to subsequent developments, can best be attributed to Hehemann and co-workers in the 1960's [6-9] where the lack of carbide precipitation but presence of retained

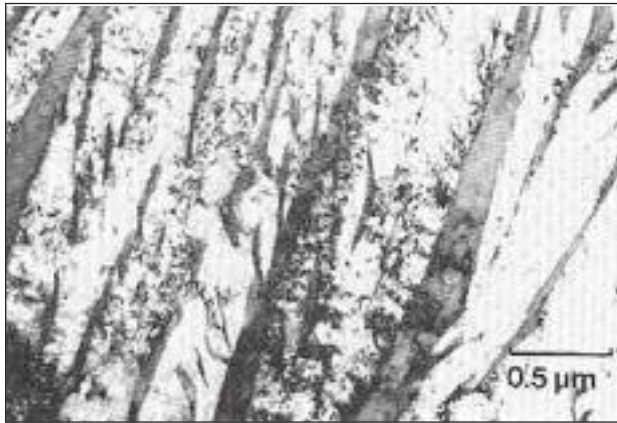
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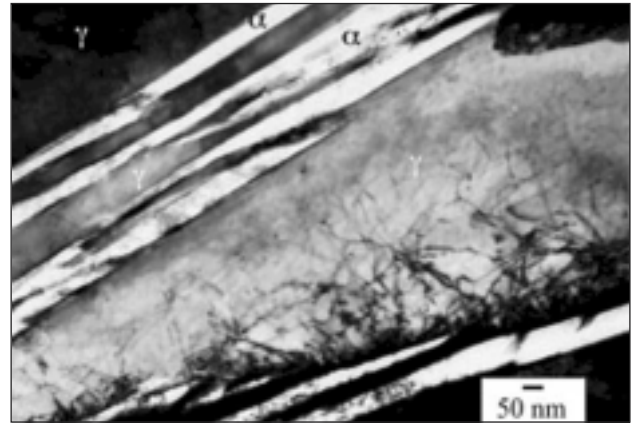
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**FIG. 1** *Electron micrograph of the interwoven bainitic ferrite and retained austenite in carbide-free bainite – retained austenite is the grey phase [5].*

*Micrografia elettronica della commistione tra ferrite bainitica e austenite residua nella bainite priva di carburi – l' austenite ritenuta è la fase grigia [5].*



**FIG. 2** *Electron micrograph of the fine nanobainite structure [28].*

*Micrografia elettronica della struttura fine nanobainitica.*

ned austenite allowed a more thorough morphological and crystallographic study of the bainite transformation mechanism [5,10]. This possibly led to the revival of commercial interest in the development of irons and steels based upon bainite, because although issues remain concerning details of the transformation mechanism [11] it is nevertheless possible to design appropriate microstructure/property combinations based upon bainitic microstructure. Notable examples include TRIP steels, and also worth mentioning because of the similarity of the basic matrix microstructure, austempered ductile iron (ADI), although it is not clear if these emerged directly from the earlier transformation studies or in parallel with them.

### Early Experimental Bainitic Steels

Some early attempts [12,13] were made to develop formability, high strength and toughness for potential exploitation in low-alloy engineering steels based upon the carbide-free bainitic microstructures developed during studies to understand the bainite reaction mechanism. Another example was a rail track steel with higher fracture toughness and lower wear rates than conventional pearlitic rail steels [14,15].

### Transformation Induced Plasticity (TRIP) Steels

Perhaps the most important development to date has been the application of carbide-free acicular microstructures to the improvement of formability at higher strength levels, giving rise to a new class of automotive sheet steels, so-called TRIP steels. The acronym TRIP – from TRansformation-Induced Plasticity – arose from the pioneering work of Zackay et al. [16,17] in the 1960's, which demonstrated enhanced ductility and formability in high-alloy metastable austenitic steels (typically, 0.2-0.3C – 8Cr – 7-24Ni – 4Mo – 2Si) due to martensite transformation during straining. TRIP-assisted behaviour in automotive steels arises from the presence of metastable retained austenite in the microstructure for low C-Mn compositions (typically, 0.2-0.4C – 1.0Mn) with enhanced Si additions of around 1.5 wt% [18-23]. In these steels, usually intercritically annealed, the microstructure is generally reported to consist of intercritical ferrite plus bainite (and maybe martensite). The bainite fraction is essentially identical to that discussed above; carbide-free regions comprising interwoven bainitic ferrite and retained austenite. The steels work in the way envisaged by Zackay and co-workers, developing enhanced

formability from the mechanically-induced transformation of the metastable austenite to martensite during processing.

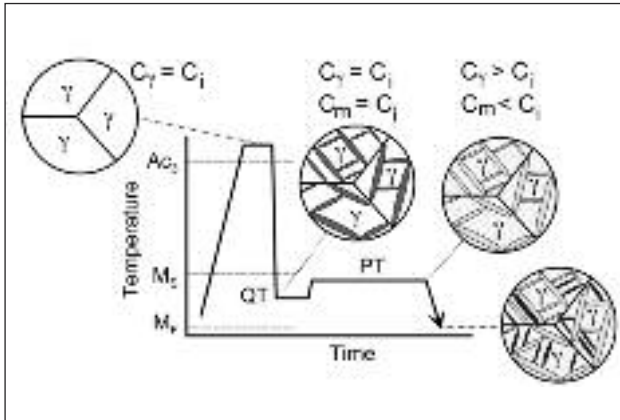
### Nanobainite Steel

The discovery of the formation of a refined carbide-free bainite at relatively high carbon concentrations (~1 wt %) after isothermal annealing for long times (up to several days) at relatively low temperatures (typically ~200°C) has led to the development of an experimental 'nanobainite' steel [24-27]. Transformation at such low temperature results in extremely thin bainitic ferrite plates ~20-30 nm thick, separated by thin retained austenite films (Fig. 2 [28]), giving hardness values ~690 HV, and yield and tensile strengths ~2.0 and 2.5 GPa, respectively.

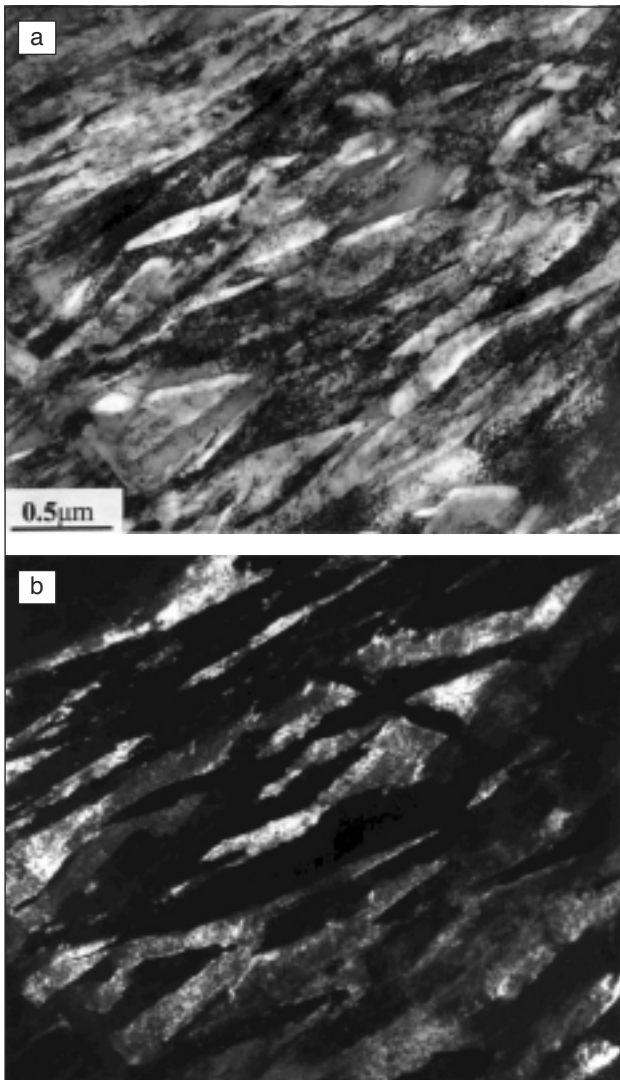
### CARBIDE-FREE QUENCHED AND PARTITIONED (Q&P) MARTENSITIC STEELS

#### The Q&P Concept

A different approach to the exploitation of steel microstructure containing retained austenite has emerged more recently, which inherits a similar philosophy to the bainitic route described above, but is based upon a martensitic microstructure [11,29-31]. This approach employs Si alloying but also adds a modified heat treatment procedure. The concept is that, firstly, an interrupted quench to a temperature (QT) between the martensite-start ( $M_s$ ) and martensite-finish ( $M_f$ ) temperatures results in untransformed 'residual' austenite (Fig. 3 [30,31]). This interrupted quench is then followed by an anneal, or so-called partitioning treatment either at, or above, the initial quench temperature (PT). With enhanced Si alloying suppressing cementite precipitation, as discussed above for carbide-free bainite formation, it is anticipated that the residual austenite will be enriched with carbon expected to escape from the supersaturated martensite phase in which it has very low solid solubility. This treatment should then produce a fine acicular aggregate of carbon-depleted and potentially carbide-free martensite laths interwoven with retained austenite stabilised by carbon-enrichment, as shown by Fig. 4 [32]). This novel heat treatment has been termed 'Quenching and Partitioning' (Q&P). It is also worth mentioning that an interrupted quench to a temperature well above ambient (effectively a partial martempering treatment) may facilitate heat treatment by minimizing any problems associated with, for example, distortion or cracking.



**FIG. 3** 'Quenching and Partitioning' (Q&P) [30,31].  
*Schema del trattamento termico 'Quenching and Partitioning' (Q&P) [30,31].*



**FIG. 4** *Electron micrographs of Q&P microstructure in AISI 9260: QT 190°C, PT 400°C; bright- and dark-field pair using an austenite reflection [32].*

*Micrografie elettroniche di microstrutture Q&P in acciaio AISI 9260: QT 190°C, PT 400°C; in campo chiaro e scuro utilizzando un punto di diffrazione dell'austenite [32].*

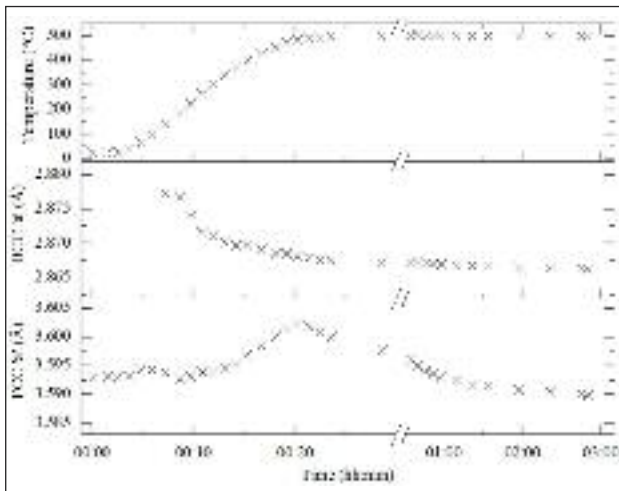
## Microstructural Evolution During Q&P Treatment

Understanding the mechanisms of microstructural evolution during the Q&P heat treatment is partly based upon experimentation and partly upon empirical hypothesis [e.g. 11,29-32]. Straightforward laboratory observations during Q&P heat treatment are frustrated by the relatively high interrupted quench temperatures involved, because certain characteristics of the heat treatment process remain inaccessible for study by conventional metallographic methods. Consequently, experimental simulation of the heat treatment has recently been carried out in alloys designed to depress the  $M_s$  temperature to facilitate more direct measurements of the sequential steps of the heat treatment process [33,34].

A 0.64C-4.57Mn-1.30Si alloy, in which the  $M_s - M_f$  range spans room temperature, has allowed a more thorough examination of the possible microstructural changes after the first quench and during subsequent partitioning. The first deduction is that the starting microstructure before the partitioning treatment is indeed a mixture of as-quenched martensite and untransformed austenite. Subsequent changes to this quenched microstructure during heating to a partitioning temperature of 500°C have been followed in real time by neutron diffraction measurements using a furnace installed on the beam line. Thus, Fig. 5 records lattice parameter and Fig. 6 microstructural parameter changes.

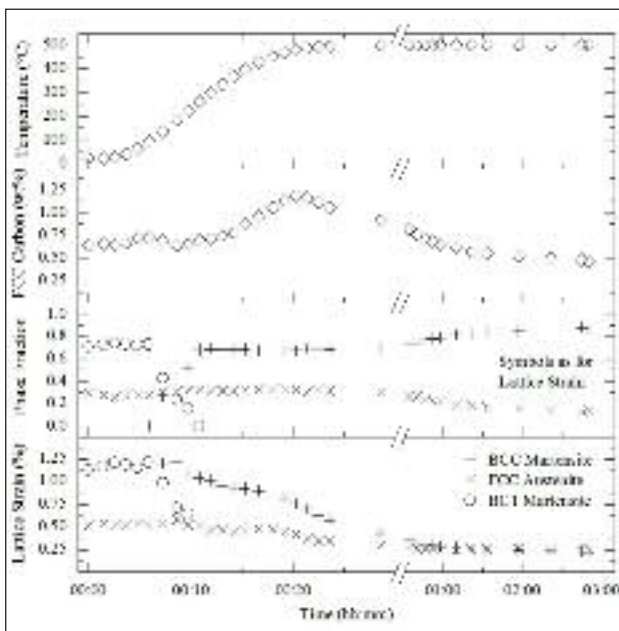
The initial BCT martensite decomposes to a BCC or low  $c/a$  ratio BCT structure, indicating release of supersaturated carbon from regular interstitial sites. Although the data collection time periods of ~1 minute are too short to determine conclusively whether carbide precipitation has occurred, published work on plain carbon steels [35] indicates that equilibrium of epsilon carbide and carbon 'trapped' in distorted interstitial sites around dislocations could be expected. The high lattice strains simultaneously measured conform to the expected high density of crystallographic defects in the body-centred lattice allowing this 'trapping' mechanism to operate. Early on in partitioning, at temperatures not much above room temperature, the equilibrium is believed to be in favour of 'trapping', while at intermediate temperatures epsilon carbide formation becomes more favourable [35] until higher temperatures where it is unstable and is replaced by cementite. However, the extent of this is difficult to assess due to its dependence upon the dislocation density, which is also dependent upon the nature of the martensite, especially the fraction of lath or twinned martensite, itself dependent upon the carbon concentration of the steel [35]. Nevertheless, it is most likely that trapping represents a very short transient stage at the higher temperatures of conventional Q&P treatment (the neutron diffraction data were collected over a relatively slow heating rate), where the alternative competing mechanisms of carbide precipitation, and in the case of a duplex microstructure and increased mobility of carbon atoms, escape to the retained austenite phase, all compete with dislocation trapping, itself diminished by recovery of the dislocation substructure (note the decreasing lattice strain with increasing temperature in Fig. 6). Figure 6 also shows the carbon concentration of the austenite eventually decreasing with time at the partitioning temperature, suggestive of carbide precipitation. Carbide peaks were subsequently detected by re-measuring at a much longer counting time after cooling the specimen back to room temperature, which confirms that carbide precipitation had indeed occurred. An example of the fine-scale nature of carbon atom clustering or carbide formation detected by very high resolution atom probe tomography is illustrated in Fig. 7 for an experimental steel with composition representative of a TRIP-assisted sheet steel (0.19C-1.59Mn-1.63Si-0.036Al-0.013P (wt.%)), austenitised at 900°C for 180 s, quenched and held at 240°C for 10 s, and partitioned at





**FIG. 5** Evolution of lattice parameters with respect to time, during heating up to, and partitioning at, 500°C (after [33,34]).

*Evoluzione dei parametri reticolari in funzione del tempo, durante riscaldamento fino a 500°C e il successivo "partitioning" a 500°C [33,34].*

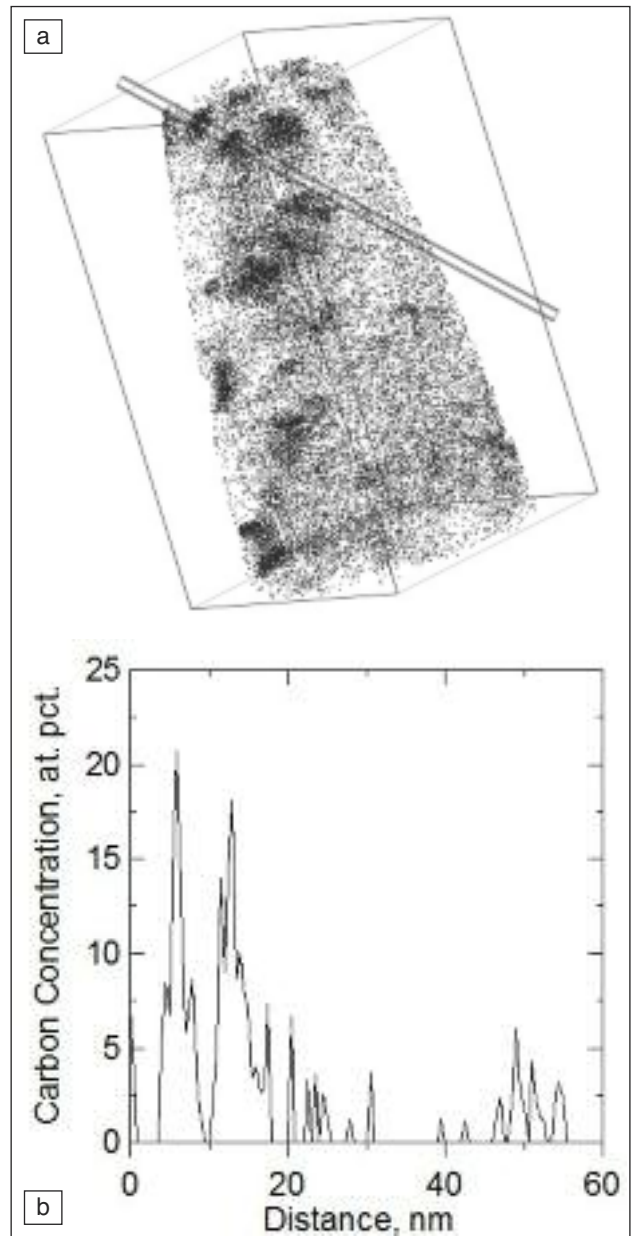


**Fig. 6** Progression of microstructural parameters with respect to time, during heating up to, and partitioning at, 500°C (after [33,34]).

*Variatione dei parametri microstrutturali rispetto al tempo, durante riscaldamento fino a 500°C e il successivo "partitioning" a 500°C [33,34].*

400°C for 30 s [36-38]. Figure 7(a) shows a three-dimensional carbon atom map reconstructed from data collected from a region approximately  $28 \times 10^4 \text{ nm}^3$ . In this particular case, the size, shape and distribution of the carbon-rich regions, and the compositions determined, were consistent with that of epsilon carbide.

Any excess carbon escaping from the martensite to the retained austenite would be expected to contribute to an increase of the austenite lattice parameter which is also reflected in the measurements in Fig. 6 and aligned with its initial increasing carbon

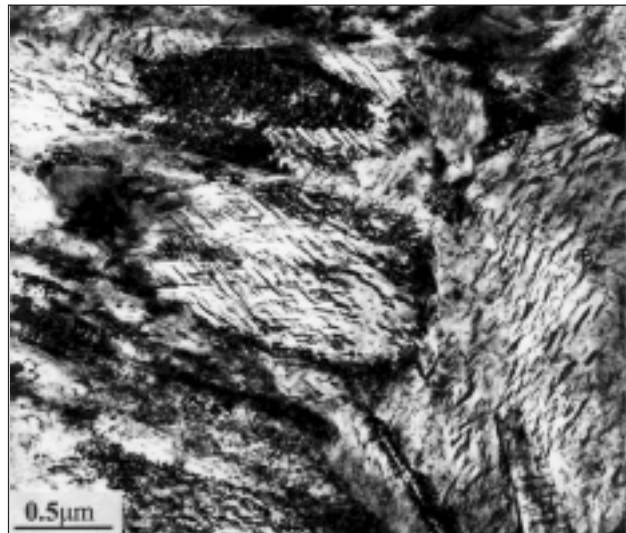


**FIG. 7** Local electrode atom probe (LEAP®) results from Q&P microstructure after partitioning at 400°C for 30 s: (a) Carbon atom map obtained from a volume  $57 \times 53 \times 93 \text{ nm}^3$  showing carbon atom clusters or carbon-containing particles, and (b) the corresponding carbon concentration profile (along the  $2 \times 2 \text{ nm}$  section indicated), with maximum peak carbon enrichment  $\sim 20$  and  $\sim 18 \text{ at.}\%$ , or  $\sim 5.4$  and  $\sim 4.6 \text{ wt.}\%$ , respectively, for each carbon rich region traversed in this example. [37]

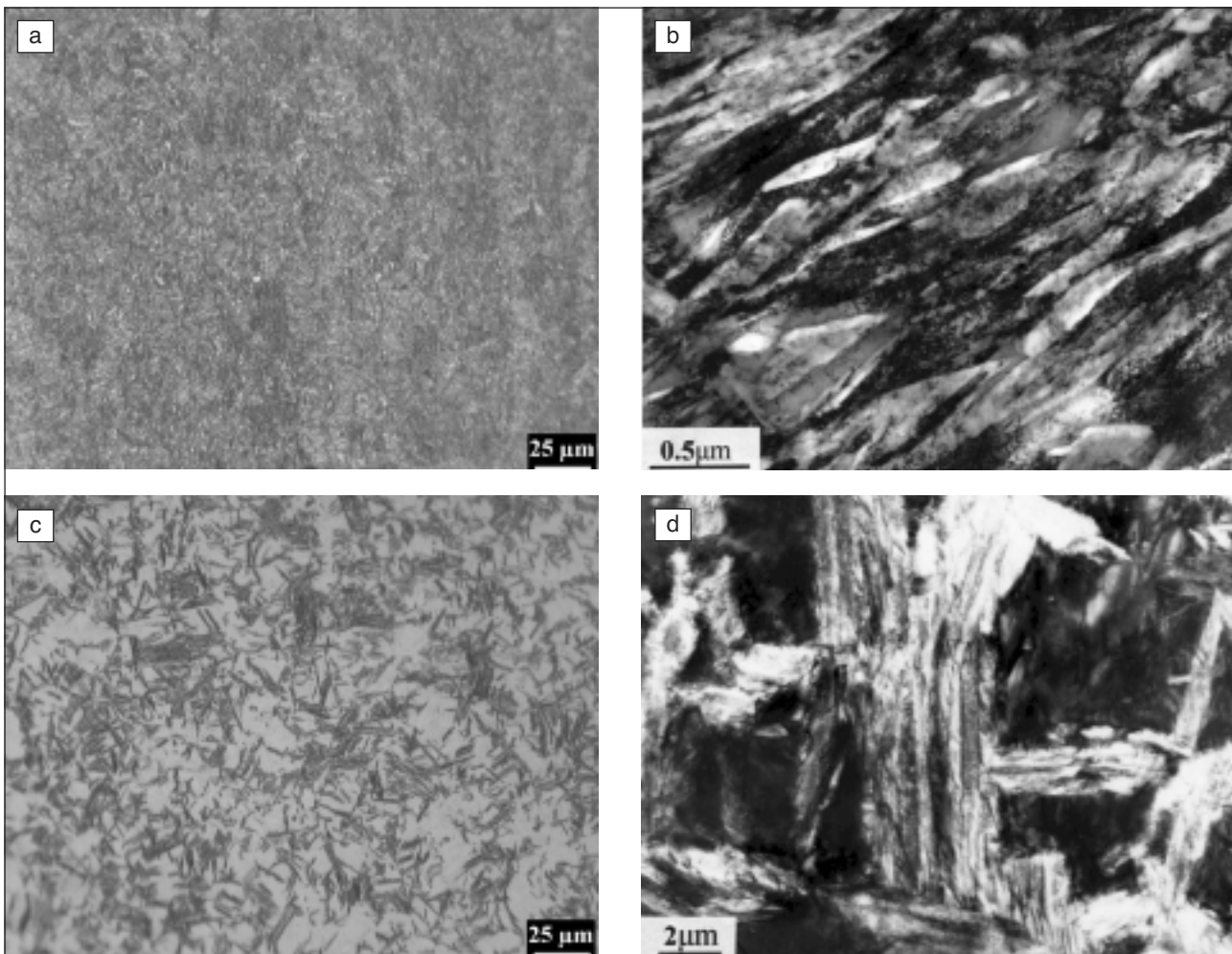
*Risultati ottenuti con sonda LEAP® (Local electrode atom probe) su microstrutture da Q&P dopo "partitioning" a 400°C per 30 s: (a) mappatura degli atomi di carbonio ottenuta da un volume  $57 \times 53 \times 93 \text{ nm}^3$  che mostra gruppi di atomi di carbonio o di particelle contenenti carbonio, e (b) il corrispondente profilo di concentrazione del carbonio (lungo la sezione  $2 \times 2 \text{ nm}$  indicata in (a)), con massimo picco di arricchimento in carbonio  $\sim 20$  e  $\sim 18 \text{ at.}\%$ , o  $\sim 5.4$  e  $\sim 4.6 \text{ wt.}\%$  in peso, rispettivamente, per ogni regione ricca di carbonio intercettata in questo esempio. [37]*

concentration. It can also be noticed that after the specimen reaches 500°C the austenite phase fraction begins to decline with partitioning time, whilst the body-centred fraction correspondingly increases. One possible mechanism for this might be austenite decomposition to bainite, previously discussed as a potential alternative competing mechanism expected at longer partitioning times [32,39].

The basic theoretical model of the Q&P process was first built upon the simple assumption that 100% of the alloy carbon content is available for stabilising untransformed austenite [30]. However, experimental results [40] soon found that upon completing the Q&P heat treatment, the measured austenite fraction was somewhat lower than would be expected if this were the case. So far, the chief explanation for this has been attributed to competition with carbide formation directly observable by transmission electron microscopy [32,37,41,42]. At the higher temperatures of partitioning it is likely that this becomes the more dominant competing mechanism than the preliminary carbon trapping by the martensite substructure, as observed above to occur at more ambient temperatures, and this is supported by customary tempering behaviour. In the lower partitioning temperature range it has been fairly conclusively shown that alloying with Si has lit-



**FIG. 8** *Electron micrograph of Q&P microstructure illustrating epsilon carbide formation [32].*  
*Micrografia elettronica della microstruttura Q&P che illustra la formazione di carburo epsilon [32].*



**FIG. 9** *Microstructures of experimental steel (0.60C - 0.95Mn - 1.96Si) given either, (a) and (b) a Q&P treatment with partitioning at 400°C for 120 s (for QT=190°C), or, (c) and (d) an austempering treatment at 400°C for 120 s. ((a) and (c) are light micrographs; (b) and (d) are bright-field electron micrographs.) [49,50]*

*Microstrutture di acciai sperimentali (0.60C - 0.95Mn - 1.96Si) sottoposti, nei casi (a) e (b), ad un trattamento Q&P con "partitioning" a 400°C per 120 s (QT=190°C), o, nei casi (c) e (d), ad un trattamento di austempering a 400°C per 120 s. ((a) e (c) sono micrografie ottiche; (b) e (d) sono micrografie elettroniche in campo chiaro.) [49,50].*





In Fig. 11 instantaneous  $n$  values versus true plastic strain are compared for two treatments: one curve, exhibiting negative slope, is for a sample quenched to a dual-phase steel microstructure, whilst the second curve, showing positive slope, is for a Q&P heat-treated sample. A positive slope of instantaneous  $n$  versus plastic strain is suggestive of TRIP behavior [55]. Thus, Q&P steel with, effectively, a tempered martensitic component, could fill a gap in this ductility/strength continuum: compared with DP and austempered TRIP steels Q&P can offer increased yield and tensile strength, whilst compared with martensitic steels, it can offer increased ductility [36,56].

It has been demonstrated that existing industrial processing lines for automotive sheet should be capable of delivering a Q&P heat treatment. Fig. 12 illustrates schematically how the Q&P heat treatment, and required time/temperature/carbide-precipitation kinetics, fit with continuous sheet processing lines [57]. In addition, comparative hole expansion tests have been conducted for austempered TRIP, bainitic (B), DP, tempered mar-

tensite (Q&T), and Q&P treated steels to assess better the automotive application [56]. As shown by Fig. 13 a better hole expansion ratio (HER) vs. tensile strength performance of Q&P steel over these alternative steel conditions was found.

### Hot Strip Steels

It may also prove possible to provide a Q&P treatment on a hot-strip mill as suggested by Fig. 14. After reduction, cooling on the run-out table is the interrupted quench (to QT), followed by cooling in the coil which can approximate to a one-step partitioning treatment (PT = QT). This implies a non-isothermal partitioning stage, but initial simulations indicate significant levels of retained austenite and high strength levels [58].

### SUMMARY

Steels with useful combinations of mechanical properties have exploited carbide-free bainitic microstructures containing carbon-stabilised retained austenite, first developed to analyse fun-

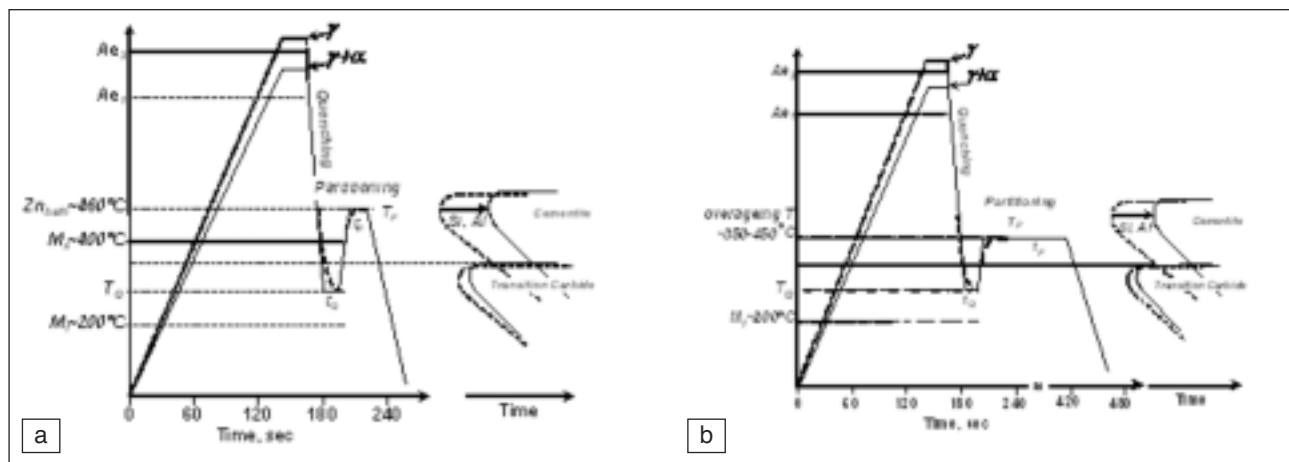


FIG. 12 Industrial potential of Q&P: (a) continuous galvanising and (b) continuous annealing lines [57].

Potenzialità industriale del trattamento Q&P: (a) linee di galvanizzazione continua e (b) ricottura continua [57].

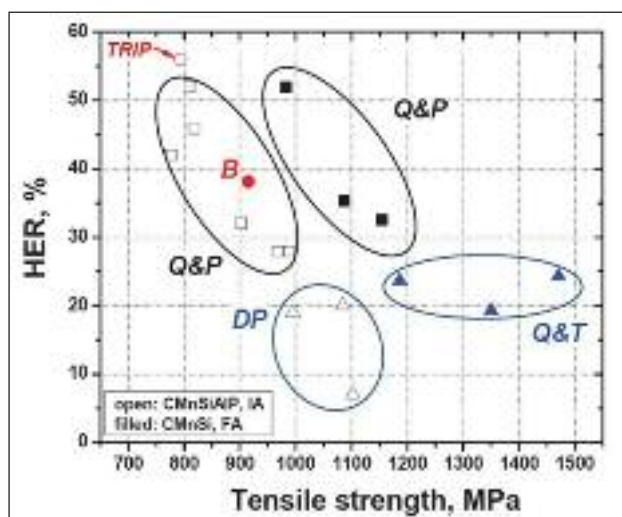


FIG. 13 Hole expansion ratio (HER) vs. tensile strength for fully austenitised (filled symbols) and intercritically annealed (open symbols) Q&P microstructures [56].

Rapporto HER (Hole expansion ratio) vs. resistenza a trazione per microstrutture Q&P completamente austenizzate (simboli pieni) e con ricottura intercritica (simboli aperti) [56].

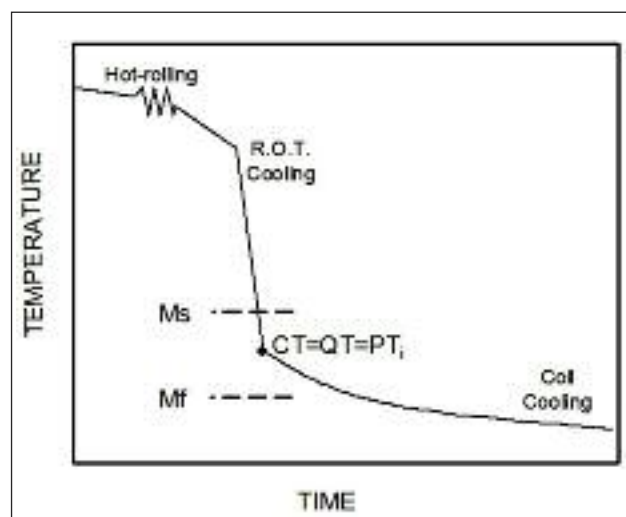


FIG. 14 Industrial potential of Q&P: hot-strip rolling [58].

Potenzialità industriale del processo Q&P: hot-strip rolling [58].

damental mechanisms of the bainite transformation. This has been followed by similar carbide-free microstructures containing retained austenite but based upon martensite, achieved by a combination of alloying and novel heat treatment procedures. This novel concept of Quenching and Partitioning (Q&P) heat treatment provides a new means of expanding applications of martensitic steels, based upon potential improvements in ductility, formability and toughness. Furthermore, preliminary considerations also indicate that the novel heat treatment cycle required could fit with existing high-tonnage processing lines, especially for cold-rolled and hot-rolled sheet.

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## Abstract

### Recenti sviluppi degli acciai con microstrutture aciculari prive di carburi contenenti austenite residua

Parole chiave: Acciaio - proprietà

Dopo un lungo periodo di progressivo miglioramento delle microstrutture degli acciai al carbonio ferritici/ perlitici mediante microalligazione e trasformazione controllata, recentemente l'attenzione si è concentrata nuovamente sulle microstrutture di forma aciculare. In particolare, un interessante progresso in questo settore è stato ottenuto con lo sviluppo - soprattutto mediante alligazione con Si - degli acciai bainitici privi di carburi (i cosiddetti "carbide-free"), in cui l'austenite residua stabilizzata dal carbonio sostituisce la cementite. Due esempi degni di nota di questi acciai sono gli acciai da lamiera "TRIP" (TRAsformazione Indotta per Plasticità) con caratteristiche avanzate, principalmente destinati ad uso automobilistico, e i futuri potenziali acciai 'nanobainitici'. Ancora più recentemente, questo concetto di sviluppare microstrutture di acciaio contenenti austenite residua arricchita di carbonio è stata ulteriormente ampliata mediante l'introduzione di nuove procedure di trattamento termico atte a sostituire la bainite con la martensite. Questo processo di non-equilibrio "quenching and partitioning", come noto, offre molti potenziali vantaggi immediati quali ad esempio: l'aumento della resistenza della struttura martensitica che risulta più protetta dai più dannosi effetti del carbonio, insieme alla possibilità di ottenere nuove proprietà dalla fase di austenite residua che può contenere potenzialmente una molto alta concentrazione controllata di carbonio.