The ideal structural steel combines high strength with excellent fracture toughness. In this paper we consider the limits of strength and toughness from two perspectives. The first perspective is theoretical. It has recently become possible to compute the ideal shear and tensile strengths of defect-free crystals. While the ferromagnetism of bcc Fe makes it a particularly difficult problem, we can estimate its limiting properties from those of similar materials. The expected behavior at the limit of strength contains many familiar features, including cleavage on \{100\}, \{111\} slip on multiple planes, "conditionally" brittle behavior at low temperature and a trend away from brittle behavior on alloying with Ni. The behavior of fcc materials at the limit of strength suggests that true cleavage will not happen in austenitic steels. The results predict an ideal cleavage stress near 10.5 GPa, and a shear strength near 6.5 GPa. The second perspective is practical: how to maximize the toughness of high-strength steel. Our discussion here is limited to the subtopic that has been the focus of research in our own group: the use of thermal treatments to inhibit transgranular brittle fracture in lath martensitic steels. The central purpose of the heat treatments described here is grain refinement, and the objective of grain refinement is to limit the crystallographic coherence length for transgranular crack propagation. There are two important sources of transgranular embrittlement: thermal (or, more properly, mechanical) embrittlement at the ductile–brittle transition, and hydrogen embrittlement from improper heat treatment or environmental attack. As we shall discuss, these embrittling mechanisms use different crack paths in lath martensitic steels and, therefore, call for somewhat different remedies.

KEY WORDS: ultrahigh strength steel; toughness; grain refinement; theory; computational science; Fe–11Ni–13Co; lath martensitic steel; ductile–brittle transition; cleavage; environmental embrittlement; hydrogen embrittlement.

1. Introduction

The ideal structural steel combines ultrahigh strength, to resist failure by plastic deformation, with high toughness, to resist failure by crack propagation. These objectives are often contradictory. Since plastic deformation is an important mechanism for relaxing stress concentrations, alloys with higher strength are, ordinarily, less tolerant of internal flaws and liable to fracture at smaller applied loads. The trade-off between strength and toughness is a recurring theme in the metallurgy of high strength steels.

In the following we shall consider the limits of strength and toughness from two perspectives. The first perspective is theoretical. Recent advances in theoretical methods and computational capabilities have made it possible to calculate the upper limits of strength for perfect crystals. Bcc Fe is a particularly difficult problem, because of its ferromagnetism, and ferritic steel is even more difficult, because it is also alloyed. However, at least some aspects of the limiting behavior of steel can be inferred from the reliable ab initio results that have been obtained for other materials. The description that emerges is necessarily tentative, but interesting. As we shall see, the expected behavior of bcc Fe at the limit of strength reproduces many of the qualitative features of the familiar behavior of high-strength steel, including cleavage on \{100\}, \{111\} slip on multiple planes, “conditionally” brittle behavior at low temperature and a trend away from brittle behavior on alloying with Ni. The behavior of fcc materials at the limit of strength suggests that true cleavage will not happen in austenitic steels. More speculatively, the results suggest that sharp cracks in steels with yield strength above about 2.5 GPa necessarily propagate in a brittle mode, though the same steels may be ductile in uniaxial tension tests, and predict a maximum value of the nanoindentation hardness of Fe at about 20 GPa.

The second perspective is practical: how to maximize the toughness of high-strength steel. At least two criteria must be satisfied to achieve high fracture toughness in high-strength steel. The fracture mode must be ductile, and ductile crack propagation must be made difficult. Our discussion of this very broad topic will be limited to the subtopic that has been the focus of research in our own group: the use of thermal treatments to inhibit transgranular brittle fracture in lath martensitic steels.

There are two basic brittle fracture modes: transgranular cleavage and intergranular fracture. The tendency toward brittle fracture is governed by the easier of the two. In a lath martensitic steel, the intergranular fracture path is along the prior austenite grain boundaries. With the single exception of some high-manganese steels, intergranular fracture is due to the accumulation of embrittling species or precipitates along the grain boundary. It can ordinarily be over-
come by a combination of good melting practice to minimize deleterious species, composition control to “getter” residual impurities into relatively innocuous precipitates, and careful heat treatment to minimize segregation to grain boundaries. Even in the case of high-Mn steels, where the weakness of the grain boundaries seems to be a property of the alloy itself, intergranular fracture can be suppressed by adding a bit of boron as a “grain boundary glue”. It follows that the maximum attainable toughness of a high-strength steel is determined by its resistance to transgranular cleavage (or “quasi-cleavage”), which is the “inherent” brittle fracture mode.

The central purpose of the heat treatments described here is grain refinement, and the objective of grain refinement is to limit the crystallographic coherence length for transgranular crack propagation. There are two important sources of transgranular embrittlement: thermal (or, more properly, mechanical) embrittlement at the ductile–brittle transition, and hydrogen embrittlement from improper heat treatment or environmental attack. As we shall discuss, these embrittling mechanisms use different crack paths in lath martensitic steels and, therefore, call for slightly different remedies.

Once a ductile fracture mode is ensured, the achievement of high toughness requires the elimination or control of void nucleation sites, a subject that is beyond the scope of the present paper.

2. The Limits of Strength in Theory

If a defect-free sample of a crystalline solid were subjected to a steadily increasing load it would deform elastically until the elastic distortion became so great that the lattice itself became unstable. At the point of elastic instability the crystal would spontaneously shear, transform structurally, or break. The elastic instability of the crystal lattice sets an absolute upper bound on the strength a material can have, the ideal strength. Recent advances in computational techniques have made it possible to calculate the ideal strength ab initio, via the pseudopotential theory.

The ferromagnetism of α-Fe makes it a particularly difficult material to study with pseudopotential techniques. The results that have been obtained to date are only approximate. However, research on simpler materials has shown that the limits of elastic stability are largely determined by crystal symmetry. These results suggest that the limits of strength of Fe can be inferred from the behavior of other bcc and fcc materials that are more accessible to the theory.

The limits of strength are interesting for at least two reasons. First, they provide values for the attainable strength. As we shall see, the estimated ideal strength of Fe is not far above strengths that have already been reached in ultrahigh strength steels and may, therefore, provide realistic guidelines for future development. Second, research into the limits of strength shows what behavior should be expected as the limit is approached. The results show that the behavior of defect-free crystals at the limited of strength is surprisingly similar to the usual behavior of high strength steels. For example: (1) Defect-free bcc metals cleave on {100} planes. (2) They shear in ⟨111⟩ directions on {110}, {112} or {123} planes under almost equal loads.

(3) Defect-free bcc metals are “conditionally brittle” at low temperature, cleaving if loaded along ⟨100⟩, but shearing if loaded in other directions. (4) Alloying elements such as Ni may inhibit brittleness at the limit of strength of bcc Fe (though at some cost in attainable strength). (5) Defect-free fcc metals (and, by inference, austenitic steels) do not cleave under tensile load, and remain ductile in the low-temperature limit.

We shall discuss these behavioral features in turn.

2.1. The Cleavage Strength of Defect-free Bcc Crystal

The low-temperature brittleness of ferritic steel is due to cleavage on {100} planes, a weakness that Fe shares with many bcc metals. Since the {100} surface in Fe is not the low-energy surface, the fundamental reason for {100} cleavage remains somewhat controversial (compare, for example, Ref. 9). However, recent ab initio calculations of ideal strength show that {100} cleavage is the natural failure mode of bcc crystals in uniaxial tension. The tensile strength of a perfect crystal of a typical bcc metal has a strong minimum for tensile load along ⟨100⟩.

The reason for the weakness in ⟨100⟩ is crystallographic. A bcc crystal that is pulled in the ⟨100⟩ direction in uniaxial tension eventually transforms into the fcc structure, as illustrated in Fig. 1. The computed tensile stress–strain curve of W is given in Fig. 2 as an example. Since the applied stress vanishes by symmetry when the strain is large enough to produce the fcc structure, it must pass through a maximum at some intermediate strain. This maximum defines the ideal tensile strength in the ⟨100⟩ direction. Since there are no comparable symmetry-induced instabilities for tension in other directions, the ⟨100⟩ direction provides the minimum tensile strength. Bcc alloys are, therefore, expected to cleave on {100} planes. Ab initio calculations give an ideal tensile cleavage stress of about 30 GPa for W (0.07E_{100}^6)^{0.10}) and an approximate value around 10.5 GPa for Fe (0.08E_{100}^6) where E_{100} is the tensile modulus in the ⟨100⟩ direction.

Once it is recognized that the ideal tensile strength is fixed by an extremum that is dictated by symmetry, its value can be estimated from a simple model that uses no quantum theory at all. If we assume constant volume, a bcc crystal is deformed into fcc by a relaxed engineering strain of 0.26 along [001] (Fig. 1). If we follow Frenkel and Orowan in assuming a sinusoidal form for the stress–strain curve, then, since the stress must vanish in both the fcc and bcc states, we have

\[
\sigma = \sigma_m \sin \left( \frac{\pi \varepsilon}{0.26} \right) = \sigma_m \sin \left[ \frac{\pi \varepsilon}{0.26} \right] \quad \text{..................(1)}
\]

where \( \varepsilon \) is the strain and \( \sigma_m \) is the ideal strength in tension.
along [001]. In the limit of small strain, \( \sigma = E_{\text{tensile}} \epsilon \), hence

\[
\sigma_m = \frac{0.26}{\pi} E_{\text{100}} = 0.08 E_{\text{100}} \quad \text{.........(2)}
\]

in very good agreement with the ab initio results.

### 2.2. The Shear Strength of a Defect-free Bcc Crystal

The ideal shear strength of a bcc metal is also set by extrema that are determined by its symmetry. Calculations of the ideal strength of bcc W suggest that the shear strength is such that, in a defect-free crystal, the common bcc metals would cleave if loaded along (100), but not if loaded in other directions. Using our estimates of the ideal strength of Fe to phrase a particular example, a uniaxial load along [100] would reach the ideal cleavage strength, 10.5 GPa, when the shear stress on the most favorable slip system, \([111](112)\), was only about 5 GPa, below the ideal shear strength of 6.5 GPa. Hence this flawless crystal would cleave in tension before deforming in shear. In this sense, Fe is inherently brittle. However, if the crystal were loaded in tension along \([111]\) or \([110]\), the shear stress would exceed the ideal strength in the \([111](112)\) slip system before the cleavage stress was reached in any \([100]\) direction. Under this load geometry, the crystal would shear rather than cleave. It follows that defect-free Fe would be ductile or brittle, depending on how it was loaded. For example, a defect-free crystal loaded in tension along \([111]\), the strongest direction, should fail in shear when the tensile load reached about 21 GPa.

This argument pertains, of course, to a flaw-free crystal under uniaxial loading. The behavior of flaw-free material in the stress-concentration field of a sharp-tipped crack is even more interesting, but is only beginning to be studied with ab initio techniques. The mechanical constraint on the crack-tip stress field has at least two effects, which counteract. First, the triaxial stress state increases the ratio of tension in the plane of cleavage, which promotes cleavage. At the same time, the planar strain state changes the strain path, which increases the cleavage stress and tends to suppress cleavage. Cleavage will also be suppressed if dislocations are spontaneously emitted from the crack tip before the cleavage stress is reached. This latter mechanism has been the subject of a number of theoretical studies in recent years. However, the inability to compute dislocation nucleation criteria ab initio has the consequence that the results are approximate and their implications are not entirely clear.

### 2.4. Alloying for Toughness in Bcc Iron

It is well known that some alloying additions, such as Ni and Mn, tend to lower the ductile–brittle transition temperature of steel. The principal reason for this is microstructural. When used in high-strength steels these elements encourage the formation of dislocated martensitic structures that combine good ductility with fine effective grain size. However, it has long been suspected that there is a chemical contribution as well, i.e., that Ni and Mn raise the inherent resistance to cleavage fracture. It is, therefore, of interest to investigate how these alloy additions might affect the ideal strength.

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**Fig. 2.** The tensile stress–strain curve for a bcc W pulled along (100) in fully-relaxed tension (uniaxial stress).

**Fig. 3.** Instability in shear in [111]. [111] shear tilts planes of atoms (equilateral triangles perpendicular to [111]) until they come into registry, as at right, creating a new symmetry.
The best fits to the polycrystalline elastic moduli give the relations:

\[ E(GPa) = 205 - 1.75x_{Ni} \]
\[ G(GPa) = 81 - 1.12x_{Ni} \]  

where \( x_{Ni} \) is the Ni content in percentage by weight.

If we assume that \( E_{100} \) and \( G_{111} \) scale with \( E \) and \( G \), Eqs. (2) and (3) have very interesting consequences. As illustrated in Fig. 4, the ideal tensile strength (i.e., the ideal cleavage stress) decreases on adding Ni. The decrease is substantial; according to Eq. (2) the ideal cleavage strength of Fe–12Ni is more than 10% below that of pure Fe. However, the shear strength decreases even more rapidly. By Eq. (3) the ideal shear strength of Fe–12Ni is more than 16% below pure Fe. The dashed line in the figure shows the resolved shear stress on the most favorably oriented member of the \( \{111\}\{112\} \) slip system when the crystal is loaded to the cleavage stress along a \( \{100\} \) direction. As Ni is added the resolved shear stress at cleavage approaches the ideal shear strength, and the crystal becomes increasingly likely to shear before cleavage. In fact, this simple model predicts that a ferritic alloy with more than about 32% Ni would be immune to cleavage.

2.5. The Limit of Strength and Ductility in Fcc Iron

Ab initio calculations of the shear strengths of defect-free Al and Cu show that they, and by inference, other fcc metals, shear most easily in \( \langle 112 \rangle \) directions on \( \{111\} \) planes, as a simple spherical-atom model would predict.\(^{21}\) The minimum shear strength is about 0.085\( G' \) in both cases, where \( G' \) is the shear modulus for \( \langle 112 \rangle \{111\} \) slip. If we assume that a similar relation holds for austenitic steel, then, with \( G' \approx 47 \) GPa, the ideal shear strength is about 4 GPa, significantly below the value for the bcc phase.

The possible cleavage planes in fcc are the \( \{111\} \) close-packed plane, which is the “quasi-cleavage plane” observed in high-N austenitic steels\(^{22}\) and the \( \{110\} \) plane, since a properly relaxed tension along \( \{110\} \) can produce the bcc structure. The \( \{111\} \) plane is improbable, since ab initio calculations for Al\(^{23,24}\) and Cu\(^{24,25}\) have found a very high tensile strength for the \( \{111\} \) direction. This result is consistent with reports of \( \{111\} \) “quasi-cleavage” in high-N steels. Careful analysis of “quasi-cleavage” in high-N steels\(^{22}\) shows that this failure mode is not true cleavage, but slip plane decohesion after significant plastic deformation.

Cleavage on \( \{110\} \) is suggested by the fact that fcc can be transformed into bcc by tension along \( \{110\} \), which creates the possibility of a tensile instability like that found in bcc.\(^{8}\) The “Bain strain” that leads from fcc to bcc via \( \{110\} \) tension is illustrated in Fig. 5. The tensile strain required to achieve the bcc structure is, in fact, relatively small, so the strength estimated by an equation like (2) would be small as well. However, to reach bcc by stretching fcc along \( \{110\} \) requires very large relaxations in the perpendicular directions. The crystal must expand isometrically along \( \{110\} \), and contract to an even greater degree along \( \{001\} \). These are unnatural relaxations that are not observed in the Poisson contractions of known fcc metals in the linear elastic limit; a relaxed tension along \( \{110\} \) does not start out on the “Bain” path and, apparently, never finds it.

Nonetheless, the \( \{110\} \) direction does seem to be the weak direction for tension in fcc. Defect-free fcc metals have very high tensile strengths when pulled along \( \{100\} \) or \( \{111\} \), but are relatively weak when pulled along \( \{110\} \). Our own recent ab initio calculations of Al and Cu give \( \{110\} \) tensile strengths of 5.2 GPa (0.07\( E_{110} \)) and 6.5 GPa (0.05\( E_{110} \)) respectively.\(^{24}\) However, the failure mode at the limit of strength in this direction is not cleavage, but shear. The crystal becomes unstable with respect to transformation into a rotated replica of itself.

Failure at 0.05\( E_{110} \) in fcc iron would correspond to a tensile stress of about 10 GPa. However, since the failure is in a shear mode, it should not produce cleavage. Moreover, the shear stress resolved on the \( \{112\}\{111\} \) slip system is about 4.6 GPa when the ideal tensile strength is reached, well above the ideal shear strength.

It follows that fcc metals like \( \gamma \)-Fe shear in preference to cleaving, and should remain ductile at all temperatures.

3. Experimental Relevance

The discussion above shows that a number of important qualitative features of the behavior of high strength steel can be inferred from its predicted behavior in the limit of strength. We now turn to the question of whether the values themselves have any physical significance. In the specific

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**Fig. 4.** The influence of Ni on the ideal cleavage and shear strengths of Fe. The lower curve shows the resolved shear stress on \( \{111\}\{112\} \) generated by the cleavage stress normal to \( \{100\} \).

**Fig. 5.** Bain strain of an fcc lattice through tension along \( \{110\} \). The crystal must expand equally along \( \{110\} \), and contract significantly along \( \{001\} \).
case of high strength steel, that question cannot now be answered. However, there are experimental indications that the predicted values of the ideal strength do provide useful guidelines to some of the properties of steel. The ideal shear strength may establish a useful limit to the microhardness of steel, while the ideal cleavage strength may set useful limits on the attainable combination of strength and toughness.

3.1. Shear Strength

Three experimental techniques have been used to explore the limits of strength in shear: tensile tests on defect-free whiskers,\textsuperscript{26} hardness tests on ultrafine-grained materials,\textsuperscript{27–29} and nanoindentation experiments on well-annealed material.\textsuperscript{30–32}

Brenner’s classic experiments on the properties of defect-free whiskers\textsuperscript{26} included tests on Fe. He reported a tensile strength as high as 13.23 GPa at ambient temperature. However, the crystal axis was believed to be (111), which suggests a critical resolved shear stress of only 3.6 GPa. His observations suggest that the tensile strength was limited by dislocation nucleation from free surfaces rather than elastic instability.

The limit of hardness in ultrafine-grained Fe has been explored by Jang and Koch\textsuperscript{27} and by Kimura, Takaki and colleagues.\textsuperscript{28,29} Jang and Koch inferred a maximum yield strength of about 9 GPa at a grain size of about 6 nm. Assuming a Taylor factor of 3, this corresponds to a shear strength of about 3 GPa. Kimura and Takaki report maximum values that are only about half this high, at somewhat larger grain sizes. In both cases the yield strength is believed to be limited by the onset of grain boundary sliding in the fine-grained microstructure.

The most direct way to access the inherent strength of bulk material appears to be nanoindentation hardness tests on well-annealed material.\textsuperscript{30} The indentation hardness of a crystal is determined by a Hertzian contact stress field with a maximum shear stress at a distance beneath the surface that is comparable to the diameter of the indenter. If the material has a relatively low defect density, and if the indenter is sufficiently small, the highly stressed volume may lie entirely within defect-free material. In this case the shear can only be relaxed by the homogeneous nucleation of dislocations or by elastic instability itself. Since the best estimates of the stress required for homogeneous nucleation place it very close to the limit of strength,\textsuperscript{33} elastic instability may well be limiting in many cases, and should, at least, provide a reasonable (and computable) value.

Nanoindentation experiments have recorded exceptionally high hardnesses in a number of metals, including the bcc metals W\textsuperscript{31} and Mo.\textsuperscript{32} Recent finite-element calculations have shown that, if the non-linearity of the stress–strain relation is taken into account, the measured nanoindentation hardnesses of W and Mo are very close to the values that are predicted on the assumption of failure at the limit of strength in shear.\textsuperscript{8,15} Unfortunately, we know of no comparable measurements on well-annealed Fe. The interesting recent work of Tsuzaki and colleagues,\textsuperscript{34} for example, was apparently done on steels with high local defect densities and produced relatively low nanohardness values in grain interiors.

Assuming an ideal shear strength of 6.5 GPa for Fe, we can estimate the maximum likely value for the nanohardness of Fe. There is some ambiguity in doing this, so we must be specific as to what we mean by hardness.\textsuperscript{30} In a normal hardness test the indenter penetrates the material quasistatically, under increasing load. In a nanoindentation test the indenter penetrates rapidly at the onset of plastic deformation, where the load drops dramatically. If we measure the hardness by the average load on the cross-section of the indentation at the point of instability, which can be calculated by finite-element techniques, the limiting microhardness of Fe should be about 20 GPa.\textsuperscript{35}

3.2. Tensile Strength

The ideal tensile strength of pure Fe is estimated to be about 10.5 GPa. This is at least twice the highest tensile strength that has been experimentally measured for steel (about 5 GPa for drawn dual-phase wire). However, much higher tensile stresses can be reached in the localized stress field of a sharp-tipped crack. It is well known from elastic-plastic finite-element analyses (FEM) that the normal stress ahead of a sharp crack can, theoretically, reach values that are more than four times the yield strength.\textsuperscript{36} It follows that tough steels with yield strength much above 2 GPa are liable to normal stresses of the order of 10 GPa.

A specific example is shown in Fig. 6,\textsuperscript{37} which shows the results of an elastic–plastic FEM calculation for grain-refined high strength steel Fe–13Co–11Ni–3Cr–1.2Mo–0.23C; (the grain-refinement procedure is described in Ref. 38). Figure 6(a) shows the stress–strain curve at 77 K. The yield strength is about 2.2 GPa. Figure 6(b) shows the max-
imeum value of the normal stress in the crack-tip stress field as a function of the applied stress intensity. As the crack blunts under the applied load, the maximum normal stress in the crack plane increases, eventually asymptoting at a value near 10 GPa. This is, of course, the theoretical number for elastic–plastic crack propagation. The best results of compact-tension fracture toughness tests on this material have it failing in a mixed cleavage-ductile fracture mode when the (calculated) maximum tensile stress is about 7 GPa.

As noted above, the ideal cleavage strength is sensitive to the stress state, and should be recalculated for the specific local stress state in the crack-tip field. Since the highly stressed material at the crack tip is inhomogeneous and plastically strained, this is not easy to do. However, if we use 10 GPa as a rough estimate of the nominal cleavage stress, the results suggest that cleavage should be the normal failure mode in fracture toughness tests on steels with yield strength above about 2.5 GPa. This inference is at least qualitatively consistent with the known behavior of ultrahigh strength steels. The best ultrahigh strength tire cord, for example, fails in a ductile mode in normal tensile tests (near uniaxial stress), but is very sensitive to the presence of flaws.

4. Achieving Strength and Toughness in Practice

The subject of this section is a very large one. We shall narrow the discussion to the area that has been the focus of research in our own group: the use of thermal mechanisms of grain refinement to achieve high toughness in lath martensitic steels, a class of steels that includes most of the successful high strength steels. We are concerned with the suppression of brittle fracture in two situations: thermal embrittlement, where brittle fracture intrudes when the service temperature falls below the “ductile–brittle” transition temperature, \( T_B \), and hydrogen embrittlement, which is the most common mechanism of environmental embrittlement in high strength steels.

Once intergranular fracture has been suppressed, the mechanism of brittle fracture is transgranular. This is the mode that must be ultimately controlled to achieve optimal properties. The mechanism of transgranular fracture in lath martensitic steels is significantly different for thermal and environmental embrittlement, so the two problems have somewhat different metallurgical solutions. However, in either case the initiating event is the fracture of a single microstructural element which, in the nominally clean and fine-grained steels of interest here, is a single “grain”, or coherent crystallographic unit. Ordinarily, cleavage initiates at local heterogeneities in the microstructure. While the local stress may approach the ideal cleavage stress at these initiation sites, the average stress is usually significantly lower, and the fracture of a single grain must be propagated into its neighbors to produce ultimate failure. The stress a cracked grain imposes on its neighbors increases with the reciprocal root of the crack length, which is limited by the “effective grain size”, \( d \). It follows that the critical value of the local stress for fracture, \( \sigma_c \), scales with the effective grain size roughly according to the Hall–Petch relation

\[
\sigma_c = K d^{-1/2}
\]

While there are elaborate theories of brittle crack propagation in real microstructures,\(^{39}\) Eq. (5) is usually adequate to guide alloy design. However, the implementation of an approach based on this simple relation requires a clear understanding of the microstructural meaning of the “effective grain size”. The “effective grain size” in lath martensitic steels depends both on the microstructure and on the embrittling mechanism.

4.1. The Microstructure of Lath Martensitic Steels

A lath martensitic steel has the characteristic microstructure shown in Fig. 7.\(^{40}\) The prior austenite grains are subdivided into packets of thin laths of martensite whose boundaries roughly parallel \( \{110\} \) planes. The internal structure of a packet is illustrated in Fig. 8. The individual martensite laths are typically less than 1 \( \mu m \) in the thin dimension, and are highly dislocated, so the microstructure appears fine-grained in optical or bright-field transmission electron micrographs. However, this steel is not necessarily fine-grained at all. The laths within a given packet (or, in some alloys, subvolumes of the packet called “blocks”) are in close crystallographic alignment, and produce simple, single-variant diffraction patterns like that shown in Fig. 8.
The lath boundaries are low-angle boundaries. The packet (or “block”) is, essentially, a single crystal of martensite.

4.2. Microstructural Mechanism of Cleavage in Lath Martensitic Steels

The microstructural mechanism of cleavage in ferritic steels is well known; ferritic steels cleave along {100} planes. It follows that, in the case of lath martensitic steels, the effective grain size is the coherence length on {100} planes, which determines the cleavage crack length. The {100} coherence length is, in turn, fixed by the packet size, or the “block” size, if the packet is subdivided into crystallographic units. The mechanism is illustrated in Fig. 9, which is a profile micrograph of cleavage fracture in an as-quenched 6Ni steel that was treated to produce very large packets and broken at 77 K. The cleavage facets follow {100} planes that are common to the laths in a packet, diverging at packet boundaries.

4.3. Environmental Embrittlement in Lath Martensitic Steels

The microstructural mechanisms of hydrogen embrittlement in lath martensitic steels are less well established. The following discussion draws on prior work in this laboratory on HY130 and 5.5Ni steel. Hydrogen embrittlement causes brittle fracture in both intergranular and transgranular modes. As in the case of thermal embrittlement, the intergranular fracture mode is ordinarily associated with chemical contamination of the boundary. Sulfur, phosphorous, nitrogen, silicon, tin and manganese have all been implicated in hydrogen embrittlement failures.

Given clean prior austenite boundaries, lath martensitic alloys like 5.5Ni and HY130 fracture in a transgranular mode when embrittled by hydrogen. The fracture mode superficially resembles the “quasicleavage” mode found in low-temperature fracture, but, as illustrated in Fig. 10, is rough and “feathery” in appearance. Crystallographic studies have shown that the dominant fracture plane is not {100}, but {110}. The {110} plane is both the dominant slip plane in bcc, and the preferred lath boundary plane. While slip may be important in some alloys, and McMahon and others have proposed modifications of the “slip-plane decohesion” model to provide a plausible mechanism, metallographic studies of 5.5Ni and HY130 steels show that lath decohesion is the dominant mechanism in these alloys.

Lath decohesion in 5.5Ni steel is illustrated in Figs. 11 and 12. Figure 11 is an optical micrograph showing the path of a transgranular crack in a quench-and-tempered specimen broken at room temperature after hydrogen charging. The figure shows cracks propagating along lath boundaries, and branching at packet boundaries. Figure 12 is a profile transmission electron micrograph of the same specimen. The fracture surface was coated with Ni prior to thinning, and is indicated by arrows in the figure. The primary crack follows lath boundaries. Secondary, subsurface cracks are also seen, also on lath boundaries.

As illustrated in Fig. 11, transgranular crack propagation in embrittled specimens of lath martensitic steel is limited by the “effective grain size”, just as cleavage cracking is. It
follows that the critical stress for fracture should obey a relation of the form (5), and that it should be possible to minimize or suppress hydrogen embrittlement by refining the effective grain size. Some successful prior work along these lines is discussed below.

But note that the effective grain size for embrittlement is the packet size in the direction of the lath boundaries, rather than across the laths on \{100\} planes. These two measures of grain size are not necessarily the same, and do not necessarily respond in the same way when the steel is processed. As discussed below, treatments that increase resistance to cleavage may actually decrease resistance to environmental embrittlement.

4.4. Refining the Effective Grain Size

The above discussion shows that it should be possible to improve toughness with respect to both cleavage and hydrogen embrittlement by refining the grain size, even though the crystallographic meaning of the “effective grain size” differs in the two cases. This approach has received dramatic new emphasis in recent years with the inauguration of large “supersteel” projects in Japan, Korea and China that are intended to develop the next generation of structural steels. The main focus of these activities is on advanced methods of thermomechanical processing. They have shown that exceptional strength-toughness combinations can be achieved in even low-alloy steels by refining grain size to the 1 \( \mu \text{m} \) range.\(^{51,52}\)

There are two major barriers to the successful implementation of these results. First, ultrafine grain size requires very high forging or rolling deformation (80% or more), and it is difficult to find practical ways of doing this. Second, it is difficult to impart large, uniform deformation through the thickness of plate steel. The first of these problems is being attacked successfully with suitable combinations of sequential deformation treatments that are applicable to sheet steels. The second remains formidable, particularly when the product of interest is high strength, high alloy plate.

There are, however, alternatives to thermomechanical methods of grain refinement that are particularly applicable to high strength plates, bars and weldments. These are cyclic thermal treatments that exploit the properties of the martensitic transformation.\(^2,40,53\) Thermal cycling treatments can be used to achieve through-thickness properties in thick plate and can be accomplished without the very substantial capital investments required for massive thermomechanical processing. Their drawback is the need for a sufficient alloy content to achieve dislocated lath martensitic structures. However, almost all of the high-strength and ultrahigh-strength steels that are candidates for advanced structural applications have alloy contents that produce lath martensite.

In fact, as we shall discuss in detail below, cyclic thermal treatments are now used in the processing of a number of modern alloy steels. While these thermal treatments were introduced for various reasons, and address a variety of metallurgical objectives, they often accomplish a grain refinement that contributes significantly to the toughness and hydrogen resistance of the alloy.

Fig. 13. Schematic phase diagram with four characteristic heat treatment temperatures indicated.

Fig. 14. (a) Bright-field transmission electron micrograph of lath martensitic steel (9Ni) after intercritical tempering. (b) Companion dark-field image; precipitated austenite appears bright.

4.5. Thermal Mechanisms of Grain Refinement in Lath Martensitic Steels

There are two generic ways to refine the effective grain size in lath martensitic steel.\(^2\) One can decrease the packet size, for example, by refining the prior austenite grain size (“grain refinement”) or, better, one can break up the alignment of laths within a packet (“packet refinement”). Both can be done with appropriate heat treatments.

The heat treatments that are commonly used to refine the effective grain size of lath martensitic steel are variations on a single theme.\(^2,53\) The alloy is quenched to martensite, then reheated to accomplish a partial or complete reversion to the austenite phase. The re-heating causes one of four characteristic reactions, which are labeled in the schematic phase diagram in Fig. 13.

(1) Tempering (t). This term designates a treatment in which the steel is held at a temperature below \( A_{11} \). The common reactions during tempering include precipitation, recovery of dislocations and equilibration of point defects. Using the symbol \( \alpha' \) for fresh martensite and \( \alpha_t \) for tempered martensite, the reaction is \( \alpha' \rightarrow \alpha_t \).

(2) Intercritical Tempering (T). This term denotes tempering at a temperature slightly above \( A_{11} \), near the bottom of the two-phase region. During this treatment a solute-rich \( \gamma \)-phase precipitates while the residual \( \alpha' \) loses solute and tempers. Most commonly, the \( \gamma \)-phase forms films along the lath boundaries of the martensite. Because of its high solute content and small size, most of the austenite is retained on subsequent cooling to room temperature, producing a microstructure like that shown in Fig. 14. The reaction is \( \alpha' \rightarrow \alpha_t + \gamma \). The precipitated austenite separates the laths within a packet. This accomplishes the “packet refinement” of the steel, in a somewhat subtle way that we shall
describe below.

(3) Intercritical Annealing (L). This term denotes a treatment just below Ac3, in the upper part of the two-phase region. An intercritical anneal forms a high volume fraction of \( \gamma \)-phase. The \( \gamma \)-phase precipitates preferentially along martensite lath boundaries, producing a microstructure of parallel laths of alloy-rich \( \gamma \)-phase and well-tempered, alloy-lean \( \alpha \). However, the \( \gamma \)-phase formed at temperatures near \( \mathrm{Ac}_2 \) is only slightly enriched in alloy content and largely re-transforms during cooling, producing a “dual-phase” structure that is a mixture of tempered and fresh martensite. The dominant reaction is \( \alpha' \rightarrow \alpha + \gamma \rightarrow \alpha + \alpha' \) There is often a small amount of residual austenite in the final microstructure.

While intercritical annealing produces a dual-phase microstructure, it does not significantly refine the grain size. The reason is that the austenite precipitated along a lath boundary has a strong tendency to retransform into the particular variant of martensite that defines the surrounding packet.\(^\text{54,55}\) However, the martensite packet is chemically heterogeneous after intercritical annealing. The \( \gamma \) is relatively rich in solute while the \( \alpha' \) is relatively lean.\(^\text{56}\) The fine-scale chemical segregation significantly affects the response to further heat treatments.

In the “spike” treatment the alloy is heated rapidly to transform it to martensite. The reaction is \( \alpha' \rightarrow \gamma \rightarrow \alpha' \). The microstructure depends on the reversion temperature and holding time. There are two limiting cases: the “step” treatment and the “spike” treatment.

In the “step” treatment the alloy is heated into the \( \gamma \)-field and held before quenching. The reverted austenite ordinarily recrystallizes if held for a reasonable time at temperatures well within the \( \gamma \)-field. If the alloy is quenched after recrystallization, but before significant grain growth has occurred, the prior austenite grain size is relatively small, and the packet size is refined.\(^\text{2,58}\)

In the “spike” treatment the alloy is heated rapidly to accomplish the transformation, then cooled immediately. The result is a dramatic “packet refinement” of the martensite; several martensite variants are mixed together on a fine scale, disrupting the crystallographic alignment of the packet and producing a very small effective grain size.\(^\text{58,59}\) The “spike” treatment is accomplished naturally in a properly designed multi-pass weldment, and is the metallurgical basis for design of ferritic weldments for ferritic cryogenic steels.\(^\text{59}\) While “spike” treatments are not ordinarily applicable to steel plate, recent research on maraging steels\(^\text{60-62}\) suggests that austenite recrystallization can be retarded in at least some alloys by adjusting the composition and austenitizing just above \( \mathrm{Ac}_3 \).

4.6. Grain Refinement of High Strength Steels

The heat treatments that are currently used to refine the grain size and control the ductile–brittle transition in high strength plate steels are combinations of the treatments described above. They include the “QT” treatment of “9Ni” steel, the “QLT” treatment of low-alloy cryogenic steels, the “LQT” treatment of “Fe–11Ni–13Co” steel and the “2B” and “2K” treatments of laboratory heats. We shall briefly describe the essential features of each.

4.6.1. The QT Treatment

For over fifty years the “workhorse” ferritic cryogenic steel has been “9Ni” steel, which has the nominal composition Fe–9Ni–1Mn–0.06C.\(^\text{63}\) It is normally processed through a QT treatment: austenitize at 800°C for 1 hr, quench (Q), then temper at 570–600°C for 1 hr (T). The Q treatment produces a dislocated lath martensite with a reasonably small effective grain size. The T treatment tempers the \( \alpha' \) and precipitates 5-10% austenite along the lath boundaries (Fig. 14).

The decrease in alloy strength that accompanies the intercritical tempering treatment decreases the ductile–brittle transition temperature \( T_B \) somewhat, but the major part of the decrease in \( T_B \) is due to the presence of precipitated austenite.\(^\text{63}\) The mechanism by which the austenite lowers \( T_B \) has been a matter of discussion. One proposal was the “blunting” action of the relatively ductile austenite phase. This mechanism was ruled out by the demonstration\(^\text{64}\) that the precipitated austenite transforms to martensite ahead of the crack tip, and is not available to blunt a fracture. Moreover\(^\text{64}\) this high-alloy precipitated austenite is not ductile, but brittle. “Transformation toughening” by the austenite is ruled out by the fact that \( T_B \) reaches its minimum value when the precipitated austenite fraction is relatively small.\(^\text{64,66}\) An increase in the austenite fraction beyond a few percent actually increases susceptibility to brittle fracture.

The interpretation that best explains the austenite effect in 9Ni and related cryogenic steels is based on the crystallography of the re-transformed austenite.\(^\text{65}\) The variant that forms on transformation under load is the one that best relaxes the local stress, and decomposes the martensite packet by introducing islands of different crystallographic orientation. The effect is to inhibit cleavage fracture and lower the ductile–brittle transition temperature. To toughen the alloy by this mechanism the precipitated austenite must be thermally stable and densely distributed through the microstructure. The thermal stability of the precipitated austenite is achieved by strong solute segregation of Ni and C at the relatively low tempering temperature\(^\text{66}\) and by the small size of the precipitated particles at the relatively short tempering times employed. The dense distribution (apparent in Fig. 14) is due to the high nickel content and the presence of a high density of lath boundary sites.

4.6.2. The QLT Treatment

Given that Ni is the costly alloy addition to 9Ni steel, there is an obvious economic incentive to reduce the nickel content. For this reason cryogenic steels of (5–6)Ni content were developed by several manufacturers\(^\text{67,68}\) and steels with Ni contents as low as 3.5Ni have been studied. These steels are processed with variants of the QLT treatment. The most thoroughly researched is 5.5Ni steel (typical composition: Fe–5.9Ni–1.2Mn–0.7Cr–0.2Mo–0.06C), which can serve as an example.\(^\text{36}\)

The intent of the QLT treatment is to achieve a dense distribution of stable austenite precipitates like that in 9Ni steel with lower Ni content. If a 6Ni steel is tempered like 9Ni steel very little austenite is formed, and that which does appear is thermally unstable.\(^\text{65}\) The austenite fraction does increase with the tempering time; tempering for 100 hr at
600°C develops about 8 vol% of precipitated austenite. But this austenite is blocky and dispersed, and does not effectively break up the martensite packets. The ductile–brittle transition remains well above 77 K.

The solution to this problem was the QLT heat treatment. In the case of 5.5Ni steel the heat treatment is: austenitize at 800°C for 1 hr (Q); intercritically anneal at 670°C for 1 hr. (L), intercritically temper at 600°C for 1 hr (T).

The microstructural changes that occur during the QLT treatment are diagrammed in Fig. 15. The Q treatment establishes a dislocated martensite structure with a small prior austenite grain size. The L step creates a fine mixture of solute-rich and solute-poor martensites; the austenite formed at 670°C contains approximately 8Ni and 3.5Mn, and retains this composition when it re-transforms. During the intercritical temper that completes the QLT treatment austenite nucleates readily along the boundaries of the high-alloy laths, which behave essentially as if they were 9Ni steel. The result is a dense distribution of stable precipitated austenite. Since the high-alloy lath boundaries are dense within the martensite packets, both the microstructure and the strength-toughness combination of QLT 5.5Ni are similar to those of 9Ni steel in the QT condition.

A similar approach was used in the design of 3.5Ni steel. However, in this case the lower Ni content makes it more difficult to assemble high-Ni laths that will form stable austenite on tempering. The successful heat treatment is designated QQQT, and involves two successive intercritical anneals, the first at a relatively high temperature to form laths of intermediate Ni content and the second at a lower temperature to form high-Ni laths within these. The final temper causes the precipitation of a dense, stable precipitated austenite. Some major steel producers also use the QLT treatment in the production of 9Ni steel to assure high quality.

4.6.3. The LQT Treatment

While the QLT treatment breaks up the martensite packet by introducing precipitated austenite, the reversed cycle, LQT, refines the packet structure directly. It is used, for example, in some heats of Fe–13Co–11Ni–3Cr–1.2Mo–0.23C secondary hardening steel to create a fine grain size that ensures ductile fracture at very high strength levels at ambient temperature.

To understand how intercritical annealing can lead to packet refinement, let an L-treated steel be given a reversion (Q) treatment. As described above, the L-treatment (intercritical anneal) creates a microstructure in which solute-rich and solute-poor laths alternate in a packet. On heating into the austenite field, both constituents of the L-treated steel revert to austenite. The low diffusivity of the substitutional species in the γ-phase prevents their homogenization. The “dual-phase” character of the alloy is preserved.

On subsequent cooling, the “dual-phase” alloy undergoes a two-step martensitic transformation. The two constituents transform at different temperatures, stressing and deforming one another. Transformation under severe mechanical constraint encourages local volumes to transform into the martensite variants that are most compatible with the local stress rather than those that continue the pattern of the martensite packet. The result is a microstructure with a very fine effective grain size.

The microstructure of this steel processed through an “NLQT” sequence is shown in Fig. 16 (“N” is an initial long-term, high-temperature anneal we used to create a very coarse starting microstructure to facilitate microstructural analysis). The prior austenite grain size and the apparent packet size are inherited from the original N-treatment and are relatively large. However, the individual laths within the packets are intermixtures of two or more martensite variants, yielding an effective grain size of less than 0.5 μm.
4.6.4. Cyclic Heat Treatments for Packet Refinement: “2B”, “2BA” and “2K”

The development of high field superconducting magnets created a need for high strength structural alloys that would remain tough in liquid helium (4 K). The alloys that are most promising for those applications are stable austenitic cryogenic steels. But it has also been shown that ferritic steels can be processed to have good toughness at 4 K.

The first successful ferritic alloy for 4K service was an Fe–12Ni–0.25Ti alloy processed through the “2B” treatment (QLQL in the terminology used above). In this treatment, reversion steps (A, Q) are alternated with intercritical anneals (B, L). The intercritical anneal decomposes the structure and increases the efficiency of the austenite reversion. Since the alloy contains Ti to getter carbon, the upper shelf toughness is naturally high. The alloy does not require tempering after heat treatment. The “2B” treatment of 12Ni–0.25Ti produces a very fine grain size whose strength-toughness combination at 4 K is equivalent to that of the better stainless steels.

An alternate to, and improvement on the “2B” treatment was the “2 K” treatment (Fig. 17) in which the alloy is heated to the intercritical temperature, held and then heated directly to the austenite field. The intent is to limit recrystallization to the tempered martensite phase to achieve a finer austenite grain size, and then take advantage of the two-step martensitic transformation of the LQ-treatment during subsequent cooling. A twice-repeated cycle of this type raises the strength-toughness combination of 12Ni steel above the 4 K trend band for conventional type 304LN stainless steels. However, examination of the fracture surface of 2 K-treated 12Ni steel at 4 K shows that the fracture is partly by cleavage, presumably in local areas in which refinement was not complete for reasons that are not yet understood. It may be possible to attain further improvements in the 4 K toughness of ferritic steel.

4.7. The influence of Grain Refinement on Hydrogen Embrittlement

The various grain refinement treatments described in the previous section are divisible into two sets: those that separate martensite laths with interlath films of precipitated austenite, such as the standard “QT” treatment of 9Ni steel, and those that refine the packet size directly, such as the “2 K” treatment of 12Ni or the “spike” treatment of ferritic weldments. Both of these treatments improve the cleavage resistance of the alloy by decreasing the mean path on the {100} planes. But it does not follow that they are equally successful in improving resistance to hydrogen embrittlement. The apparent mechanism of transgranular embrittlement is interlath cracking, and the precipitation of interlath austenite does not refine the effective grain size in this direction.

In fact, prior work suggests that intercritical tempering actually reduces hydrogen resistance in alloys that embrittle in the transgranular mode. The mechanistic research was done on 5.5Ni (6Ni) steel. In the QT condition, with essentially no austenite, this alloy has a yield strength of 740 MPa and a fracture toughness of 330 MPa√m at ambient temperature. In the QLT condition the lath boundaries of the alloy are densely decorated with precipitated austenite (Fig. 18). The yield strength is about 590 MPa and the fracture toughness is about 360 MPa√m. More importantly for the purposes for which that alloy was designed, its cleavage resistance is very high and its ductile-brittle transition is below 77 K.

When the alloy is charged with hydrogen, it embrittles in a transgranular mode. Its yield strength increases slightly, but its fracture toughness drops significantly, to 180 MPa√m for the QT condition, and to 100 MPa√m in the QLT condition. The significantly lower toughness of the QLT alloy shows that embrittlement is significantly worse when austenite is precipitated along the lath boundaries.
The parent reason for this behavior is suggested by the profile TEM fractograph shown in Fig. 19.

As illustrated in Fig. 19, the fracture mode in the alloy is interlath. In addition, there are frequent secondary cracks in the lath boundaries just beneath the fracture surface. These invariably emanate from islands of fresh martensite, which are the transformation product of the precipitated austenite formed during the QLT treatment. It appears that the transformation of the austenite contributes to the embrittlement of the lath boundary.

The probable mechanism by which the martensite transformation contributes to embrittlement is the “wedging” effect of the transformation strain. The martensitic transformation involves a positive volume change, with an associated shear. The transformation of an island of austenite along the lath boundary hence produces a mechanical “wedge” that acts to split the boundary open. If the boundary is already embrittled, this new mechanical load can produce a brittle, interlath fracture of the sort commonly found in profile fractographs of embrittled QLT-treated 6Ni steel.44)

A very different result is obtained when a similar alloy, Fe–12Ni–0.25Ti, is “packet refined” by repeated rapid “spike” cycles into the austenite field. Figure 20 illustrates the results.42) If the alloy is austenitized and quenched before hydrogen charging it is significantly embrittled in a transgranular mode, as shown in Fig. 20(b). The alloy can be embrittled further by adding a temper embrittlement treatment, 300 hr at 450°C before hydrogen charging. In this case the fracture mode is intergranular (Fig. 20(a)). A single “spike” reversion into the austenite field produces the rather mixed fracture surface shown in Fig. 20(c). The toughness is higher, but the alloy is still quite brittle. However, a second “spike” reversion produces the highly refined microstructure shown in Fig. 21. In this microstructure, the alloy is virtually impervious to hydrogen. Even

![Fig. 19. Transmission electron fractographs of 6Ni steel in the condition after embrittlement by hydrogen. The interlath fracture is marked by arrows in the upper figure. Secondary interlath cracks appear below the fracture surface. These emanate from islands of fresh martensite in the lath boundaries.](image)

![Fig. 20. Scanning electron fractographs of 12Ni–0.25Ti steel after hydrogen charging. (a) Intergranular embrittlement caused by quenching followed by temper-embrittlement at 450°C. (b) Interlath fracture in the as-quenched condition. (c) Interlath fracture after a single “spike” reversion to austenite. (d) Ductile fracture of sample packet-refined by sequential “spike” treatments and tempered for 300 hr at 450°C.](image)

![Fig. 21. Transmission electron micrograph showing the microstructure of a Fe–12Ni–0.25Ti alloy given two “spike” reversions to austenite. The diffraction patterns and dark-field micrographs show that the microstructure is broken up into submicron elements separated by high-angle grain boundaries.](image)
when it is given 300 hr temper embrittlement at 450°C before hydrogen charging, the alloy fractures in a fully ductile mode, as shown in Fig. 20(d).

As these results illustrate, the heat treatment of lath martensitic steels that are to be used in aggressive environments should be chosen to defeat the actual mechanism of failure. If the failure mode is transgranular hydrogen embrittlement, packet refinement to ultrafine grain size lead to exceptional resistance, while the precipitation of interlath austenite does not help, and may even be harmful.

5. Conclusion

There is reason to believe that we are, collectively, beginning to understand the real limits on the achievable properties of steel as we continue to make progress in achieving them.

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