TWO METHODS FOR PROCESSING AN ULTRAFINE FERRITIC GRAIN SIZE IN STEELS AND THE THERMAL STABILITY OF THE STRUCTURE

LONGXIU PAN

Faculty of Technology,
Department of Mechanical Engineering,
University of Oulu

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Faculty of Technology, University of Oulu, P.O.Box 4000, FIN-90014 University of Oulu, Finland,
Department of Mechanical Engineering, University of Oulu, P.O.Box 4200, FIN-90014 University of Oulu, Finland
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Abstract
In this thesis, methods to process ultrafine ferritic (UFF) structures in steels, i.e. grain sizes below about 3 μm have been investigated. It is shown here, in accordance with the results in the literature, that a steel with a UFF grain size can be obtained by two methods, more or less convenient to mass production: deformation-induced ferrite transformation from fine-grained austenite (the DIF route) and the static recrystallization of various heavily cold-worked initial microstructures (the SRF/SRM route).

In the present work, the influencing factors in the processing of UFF structure in the DIF route have been systematically studied in four low-carbon steels: one C-Mn steel and Nb, Nb-Ti and Nb-high Ti microalloyed steels. A high strain, a low deformation temperature close to Ar3 and a fine prior austenite grain size are beneficial to promote the formation of UFF grains. Especially by using complex pretreatments to refine the prior austenite grain size, cold rolling, repeating the low-temperature reheating cycle or using martensitic initial microstructure, a UFF grain size can be obtained in these steels at the strain of 1.2 (70% reduction) at 780 °C. By controlling the cooling rate, the type of the second phase can be adjusted.

When using the static recrystallization route, it was found that UFF is difficult to obtain from a single-phase ferrite, but it is relatively readily obtained from deformed pearlite, bainite or martensite, especially in high-carbon steels with 0.3–0.8%C. In deformed pearlite, the cementite lamellae fragmented and spheroidised in the course of heavy deformation can provide numerous nucleation sites by the particle stimulated nucleation mechanism and retard the subgrain and recrystallized grain growth. Nucleation and retardation of grain growth are effective also in deformed bainite, martensite or high-carbon tempered martensite, as discussed in detail in the work.

The thermal stability of UFF grained steels was tested and found to be generally excellent, but it varies depending on the processing method. The UFF structure obtained by the SRM route has a thermal stability somewhat weaker than that of the DIF route. For a given steel, UFF grains may show different grain growth modes, related to the dispersion of second phase particles. In the DIF structure, abnormal grain growth occurs at 700 °C after about 2.5 h, while in the SRM structure, normal grain growth takes place slowly at 600 °C. Carbides on the grain boundaries seem to play an important role in inhibiting grain coarsening. No coarse-grained zone was formed at the HAZ of electron beam or laser welded seams, as performed at low heat inputs (up to 1.5 kJ/cm) on thin strips. The hardness even increased from the base metal towards the HAZ and the weld metal in all seams as an indication that they were hardened during the rapid cooling.

Keywords: bainite, deformation, electron beam welding, high carbon steels, laser welding, low-carbon steels, martensite, microalloy steels, pearlite, static recrystallization, tempered-martensite, thermal stability, ultrafine grain size
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List of symbols and abbreviations

Latin letters
A electric current
A constant (p.18)
ARB accumulative roll-bonding
ASTM American society for testing and materials
A$_1$ phase transformation temperature where ferrite begins to transform
to austenite
A$_3$ phase transformation temperature where austenite begins
to transform to ferrite
A$_{c1}$ temperature at which ferrite begins to transform
into austenite in heating
A$_{c3}$ temperature at which all ferrite has transformed
into austenite in heating
A$_{e3}$ phase transformation temperature from austenite to ferrite at equilibrium
A$_{r3}$ temperature at which austenite begins to transform
into ferrite during cooling
B constant
C a material parameter depending on mobility
CEV carbon equivalent value
CGHAZ coarse-grained heat-affected zone
d grain size
d$_{EFF}$ effective grain size
d$_0$ initial grain sizes before growth
D$_{\alpha}$ ferrite grain size
D$_{\gamma}$ austenite grain size
DBTT ductile-brittle transition temperature
DIF deformation-induced ferrite; strain induced dynamic transformation of ferrite from austenite

D_{LIM} grain size at which the normal grain growth will cease

D_N grain size after primary recrystallization by particle stimulated nucleation

DRV dynamic recovery

DRX dynamic recrystallization

DRF dynamic recrystallization of ferrite

d_Z Zener limiting grain size

e elongation

FGHAZ fine-grained heat-affected zone

EBSD electron backscattered diffraction

ECAP equal channel angular pressing

EL elongation

F/P ferrite/pearlite

F_v volume fraction of particles

HAZ heat-affected zone

HSLA high-strength low alloy steel

HV Vickers pyramid hardness

ICHAZ intercritical heat-affected zone

IF interstitial-free steel

k_y grain size strengthening coefficient

K-S Kurdjumov-Sachs orientation relationship

LYP lower yield point

MAG metal active-gas arc welding

n grain size exponent

OM optical microscope

PSN particle stimulated nucleation

Q quenched

QT quenched and tempered

r small particle mean radius

R_{p0.2} yield strength at 0.2% plastic strain

SEM scanning electron microscope

SHT Sumimoto High Toughness

SIDT strain-induced dynamic transformation

SRF static recrystallization of deformed ferrite in annealing

SRM static recrystallization of deformed martensite in annealing

SRP static recrystallization of deformed pearlite

SUF surface layers with ultra fine grains
t time
TEM transmission electron microscope
T_m melting temperature
TMCP thermomechanical controlled processing
Trs transition temperature
TS tensile strength
T cooling rate
t_{8/5} cooling time between 500 and 800 °C
Uf/C ultrafine ferrite/cementite
UFF ultrafine ferrite
ULC ultralow carbon
V voltage
WTO World Trade Organization
WM weld metal
YS yield stress

Greek letters
\( \alpha \) ferrite
\( \alpha \) a constant depending on chemical composition (p.35)
\( \beta \) a constant representing the degree of resistance of grain boundaries
to the brittle crack propagation
\( \gamma \) austenite
\( \Delta K \) stress intensity range
\( \sigma_0 \) friction stress
\( \sigma_y \) lower yield stress
\( \sigma_{yc} \) cyclic yield stress
\( \sigma_{0.2} \) yield stress at 0.2% elongation
\( \varepsilon \) retained strain
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References
1 Introduction

1.1 Recent trends in steel development

Steel materials have the features of low cost, large-scale production, easy processing, reliable performance and practically 100% recyclability. Steel is an irreplaceable structural material widely used for many decades for building constructions, bridges, automobiles, ships, and so on. Any improvements in its performance have a great influence on society and the economy. For instance, a higher strength means weight and energy savings, less air pollution, cost reduction, etc.

Today steel industries are facing new challenges to cope with. Firstly, the developments of information and communication technologies and the open trade market under the WTO trading system will drive steel companies into an intensive competition environment that urges them to develop new innovation technologies for their survival. Secondly, because the steel industry is energy- and resources-intensive in its nature, it is inevitable for steel companies to develop energy-efficient and environmentally friendly processes due to the situation that energy and environmental issues will come to the front. Finally, considering the high technological level in the future, the steel products used up to this time will not be able to sufficiently meet the demands of high-standards in the next generation.

High-strength steels with excellent impact toughness find many applications, especially in heavy structures, where they can offer economical benefits as reducing both material and fabrication costs. Therefore, high expectations are set on structural ferritic steels with fine or even ultrafine grain sizes, which have the potential to provide superior performance compared to conventional steels.

Several strengthening methods have been found to improve mechanical strength, such as solid solution strengthening, precipitation hardening, grain size refinement, transformation hardening, dislocation hardening, and texture strengthening. Among these methods, only grain size refinement is the strengthening mechanism that can also improve impact toughness. It has been proved that, when the grain size of ferrite is refined to 1 µm, the yield strength can increase by almost 350 MPa, compared to a steel with the 5 µm
grained ferrite that is regarded as the grain size limit in conventional controlled rolled steels [1].

A considerable refinement in the grain size can be achieved economically in a hot rolling mill in thermomechanical controlled processing (TMCP) rather than through any additional heat treatment. In TMCP, expensive alloy additions and heat treatments are avoided, and due to a lean composition the steel has also a good weldability [2].

The relationship between the yield stress (or more generally strength) and the grain size is well known as the Hall-Petch equation, Eq. (1):

\[
\sigma_y = \sigma_0 + k_y \cdot d^{-1/2}
\]

where \(\sigma_y\) is the lower yield stress, \(\sigma_0\) is the friction stress, \(k_y\) is the strengthening coefficient and \(d\) is the grain size. This relationship is schematically plotted in Fig. 1 that also shows the ranges of ferrite grain sizes typically obtained in two industrial processing routes [3,4]. It can be seen that a yield strength level of 800 MPa could be expected to be attained with a by grain size of 1 \(\mu\)m. Fig. 2 displays an example, a Japanese ultrafine grained C-Si-Mn steel and its typical stress-strain curve together with a stress-strain curve of a conventional C-Mn steel [5]. A highly enhanced strength of the ultrafine grained steel is obvious. The similar trend is also valid between the impact toughness and the grain size, plotted in Fig. 1. Accordingly, if reducing the grain size from 10 \(\mu\)m to 1 \(\mu\)m, the transition temperature decreases by 250 K.

![Fig. 1. Relationships between the ferrite grain size and the strength and toughness of ferritic steels as processed via different routes (STX21 = a Japanese project Ultra-Steels, Structural Materials Xs for the 21st century) [1].](image-url)
Fig. 2. A Japanese ultrafine grained C-Si-Mn steel and its stress-strain curve compared to that of a conventional steel [5].

In TMCP, the finest grain size attainable is at an order of 4 µm, in routine production 7-10 µm [2], and further refinement is not possible by this way. In the past 25 years, only limited progress has been made commercially in developing more efficient grain refining progressing techniques, but in the recent last five years or so, all over the world, in Japan, Korea, China, Australia and Europe, much effort has been directed to develop new manufacturing methods to obtain microstructures with an ultrafine ferrite (UFF) grain size (0.2~2 µm). Really, some achievements have been reached at the laboratory scale by various processing techniques, such as the phase transformation from austenite to ferrite under a strong magnetic field [6, 7], severe plastic straining in the equal channel angular pressing/extrusion [8-12], torsional deformation [13], accumulative roll-bonding [14], and thermomechanical rolling under special conditions [1, 6, 7, 13, 14, 15, 16]. For the mass production of metal products, hot rolling with heavy deformation can be expected to be the most practical and economical way for the manufacturing the UFF steels. The other potential routes, such as static recrystallization of ferrite after cold rolling of pre-refined ferrite (SRF) and dynamic recrystallization of ferrite (DRF) have also been reported to gain some success [17, 18]. Using the above-mentioned methods, the 4-5 µm grain size limit has been broken in laboratory and pilot mill experiments. For instance, 2-3 µm grain size has been achieved at CRM in Belgium on the hot strip mill with ultrafast cooling [19], and 2 µm in certain steel grades by rapid annealing of cold rolled strips [20]. In Japan, Nippon Steel has introduced a plate product to the market with a 2-3 µm grain size in the surface layer [21]. At Nakayama Steel Works at Osaka a new hot strip mill has been built, where UFF grained strip steel can be produced with a grain size of about 3 µm in a 2 mm strip [22].
1.2 Thermomechanical processing and grain size

TMCP, i.e. controlled rolling and accelerated cooling, has been utilized for more than 20 years now for the manufacturing of high-strength low-alloy steel (HSLA) plates and sheets with a yield strength up to 500 MPa (sheets up to about 700 MPa) and excellent impact toughness and weldability. The microstructure of these steels is essentially ferritic, with a grain size of 4 µm at the minimum (see Fig. 1). The controlling factors on ferrite grain size are well known and reported in open literature, and are connected with the austenite grain size in the reheating process, recrystallization during the rough rolling stage, accumulation of strain in the finishing rolling stage in the non-recrystallization regime of austenite, and the austenite decomposition in the accelerated cooling stage, see e.g. [2]. Similar phenomena are to some extent present also in the formation of UFF microstructures so that they are briefly discussed in the following.

1.2.1 Strain-free austenite

In the case of the transformation of ferrite from recrystallized, strain-free austenite, only prior austenite grain boundaries provide the nucleation sites for the ferrite phase. The transformed ferrite grain size ($D_\alpha$) is affected by the cooling rate and the prior grain size of austenite ($D_\gamma$), and the type of nucleation sites (grain corners, edges or boundary faces). A typical experimental relationship between $D_\alpha$, the cooling rate ($T$) and $D_\gamma$ in an Fe-0.15C steel is as below:

$$D_\alpha = 5.7T^{-0.26}D_\gamma^{0.46}$$

(2)

where $T$ is the cooling rate in °C/s, and the grain sizes are in µm [23].

However, for the cooling rate a maximum exists, for if it is exceeded, the austenite will transform into other phases than ferrite, i.e. bainite or martensite. Therefore, the refinement of the ferrite grain structure is achieved mainly through the refinement of the austenite grain size. Although the ferrite grain size decreases with decreasing austenite grain size, it reaches a limiting value of around 10 µm, when the austenite grain size becomes about 10 µm, as shown in Fig. 3. Hence, there is a limit in attaining the ferrite grain refinement through the $\gamma/\alpha$-transformation from recrystallized, strain-free austenite [24].
1.2.2 Deformed austenite

In the plastic deformation in the non-recrystallization temperature regime, the austenite grains become elongated and they contain deformation structures, deformation bands and twins inside the grains that highly increase the number of nucleation sites and the nucleation rate of ferrite. A quantitative relationship has been proposed by Bengochea et al. [25], among others, between the ferrite and austenite grain size, cooling rate, and the retained strain in the austenite as follows:

\[
D_\alpha = \left\{ 4.5 + 3T^{0.5} + 13.4 \left[ 1 - \exp(-0.015D_\gamma) \right] \right\} \times (1 - 0.5\epsilon^{0.47}) \tag{3}
\]

where \(\epsilon\) is the retained strain.

The dependence of ferrite grain size on the cooling rate with increasing amount of retained strain in the austenite for the prior austenite grain size of 30 µm is shown in Fig. 4a. The dependence of ferrite grain size on the austenite grain size under accelerated cooling conditions and with retained strain is plotted in Fig. 4b. It can be seen that the cooling rate has only a small influence, and the retained strain must be high (> 1) to obtain a fine ferrite grain size even from a fine-grained austenite.
Hodgson et al. [26] proposed another equation:

$$\frac{1}{D_\alpha} = \frac{A\sqrt{T}}{D_\gamma} + B\varepsilon \frac{2}{3}$$

where $A \approx 4.0$, $B \approx 0.15$. Accordingly, the effect of retained strain on the ferrite grain size is shown in Fig. 5. A fine ferrite grain size can only be obtained from fine-grained austenite and at high retained strains and cooling rates.

In recent years, several research groups have reported that they have achieved a ferrite grain size below 5 µm in low-carbon microalloyed and plain carbon steels using laboratory-scale thermo-mechanical processing methods, e.g. [1, 15]. At Sumitomo [27],
NKK [28] and POSCO [1] companies, a ferrite grain size of 2 µm has been attained. At CRM in a hot strip mill with an ultrafast cooling unit, a ferrite grain size of 2~3 µm has been successfully produced [19]. Nippon Steel has introduced a plate product to the market with a 2-3 µm grain size at the surface layer in 18-50 mm thick plates [21, 29]. Yada et al. [30] produced low-carbon 0.13C-0.32Si-1.07Mn steel (Note: in this thesis, the concentration values are in mass-%, if not otherwise mentioned) strips of 3.5 mm in thickness with a mean grain size of about 3 µm. According to single-pass deformation experiments, a reduction of 80% was necessary to achieve this fine grain size [31]. The deformation could also be given in two or more passes, if the interpass time was between 0.2 and 8 s.

1.3 Routes to obtain UFF microstructure

There are several ways that can refine the grain size of ferrite smaller than obtained in the conventional TMCP, i.e. to the ultrafine scale (viz. order of 1-3 µm or less), even though most of them are quite exotic techniques.

1.3.1 Equal channel angular pressing

The equal channel angular pressing (ECAP) technique was developed about 20 years ago to introduce severe plastic strain into material without causing any change in the cross-sectional area and extensively used for processing ultrafine grain sizes especially in aluminium alloys in the last few years [32, 33, 34, 35]. Severe plastic straining is achieved in ECAP by pressing the sample through a die as shown in Fig. 6.

The sample is machined to fit within a channel which passes through the die in an L-shaped configuration (various angles can be used). Passing through the angle, the sample will undergo straining in shear. Because of no change in the cross-sectional dimensions, the process can be repeated as many times as wanted in order to achieve very high total strains. It is also possible to rotate the sample between consecutive pressing passes so that different shearing systems are activated.
Most of the work on steels has emerged in recent years [8, 10, 11, 12]. The ECAP technique has been applied to pure iron [10], IF steel [10] and commercial 0.15%C steel [11]. Grain size of 0.2 µm in the IF steel and nanometer grains in the low-carbon steel have been reported [12].

1.3.2 Powder metallurgy

There are two basic types of fine-grained alloys, which can be produced by powder metallurgy [36]. One type has an oxide phase dispersed through the metallic matrix, where the oxide constrains the grain growth. The other type is produced by the consolidation of atomized alloy powders. For instance, Belyakov et al. [37] have investigated microstructure evolution in iron powder under mechanical milling and consolidating rolling.

1.3.3 Thermal cycling

The prior-austenite grain size of steels can be refined to ASTM 14.5 (≈ 2 µm) by repeated cycles of austenitizing and transformation [38]. Several factors determine the final grain size such as the steel composition, starting microstructure, heating rate through the transformation range, peak temperature, the time that the steel is held above the $A_3$ temperature, and the number of cycles of rapid heating and cooling.
1.3.4 Accumulative roll-bonding

In the accumulative roll-bonding process (ARB), as shown in Fig. 7, a strip is placed on another strip of the same thickness and rolled to a 50% reduction [34, 35]. To ensure a good bonding, the surfaces of the strips are cleaned both mechanically and chemically. The material is then sectioned into two halves in length, stacked and rolled together. The whole process can be repeated again and again.

Fig. 7. Accumulative roll-bonding (ARB) process to manufacture ultrafine grain size in metals [39].

The process is conducted at a warm rolling temperature in a ferrite regime below the recrystallization temperature to accumulate strain in the ferrite. It has been reported that ultrafine grains of 0.4-1 µm have been obtained in Ti-stabilized IF steels [13]. Based on the TEM analysis, it is suggested that the formation of ultrafine grains occurs by grain subdivision under intense plastic straining and by the grain boundary formation aided of the recovery process.

1.3.5 Dynamic recrystallization of ferrite

It has generally been considered that dynamic recovery (DRV) is the only restoration process during hot-deformation in the ferrite phase. The main barrier to the occurrence of dynamic recrystallization (DRX) is the presence of interstitials such as C and N. However, DRX in ferrite has been reported in an IF-steel by Najafi-Zadeh et al. [40] and Jonas [41]. It is thought that DRX can occur in two instances, under warm rolling conditions at the temperatures of 600 - 850°C at strain rates between 20 - 200 1/s and in the second case at very high strains (exceeding 3) that can be reached by the combination of heavy rolling reductions and strain concentration in the soft ferrite phase in rod or bar mills [2, 42, 43]. Azushima et al. [44] obtained DRX in multi-axial deformation.
The recrystallized ferrite grain size decreases with the decreasing finishing pass temperature. Najafi-Zadeh et al. [45] reported that in DRX of ferrite in a Ti-IF steel, a ferrite grain size of 1-3 µm can be obtained depending on the rolling conditions. Matsubara et al. [46] showed that DRX of ferrite occurs heterogeneously and an increase in the strain rate can refine the ferrite grain size. Ti and/or Nb addition can promote DRX by enabling strain accumulation to occur.

1.3.6 Deformation-induced ferrite formation

It had been confirmed that ferrite can form from austenite during deformation at high strains and low deformation temperatures ("warm deformation"), in a process termed deformation-induced ferrite formation (DIF) [1, 6, 15, 16] (some researchers call it strain-induced dynamic transformation, SIDT [47, 48]. Priestner and Ibraheem [48] used this method as a modification of conventional TMCP route (they called it transformational grain refinement) and showed that a grain size of 1 µm could be obtained in a Nb-bearing steel. Fig. 8 schematically shows this processing route and some variables that affect the final grain size achieved.

![Fig. 8](image)

Fig. 8. Schematic depiction of a processing route for UFF structure consisting of transformation refining the prior austenite grain size (prior tempering and cold working), heavy deformation of austenite and accelerated cooling [48].

The detailed mechanism of DIF is not very clear yet. As the strain exceeds the critical strain for DIF, it can start and DRX will be retarded. There are evidences that the ferrite is formed dynamically during the hot deformation, not subsequently [47, 50, 51]. First, the deformation resistance during the hot working is much lower than that expected for the austenite, and the decrease is observed to be proportional to the fraction of ferrite formed. The strength of low carbon ferrite is generally accepted to be considerably smaller than that of austenite at the same temperature. Secondly, even very high quenching rates can not suppress the formation of the ferrite, which amounts to over 90% in volume fraction.
when large strain is applied to a specimen. Thirdly, the ferrite is unstable above the equilibrium start temperatures of the proeutectoid ferrite, $A_{e3}$, decreasing in volume fraction with time during isothermal holding following the deformation, while it is stable below $A_{e3}$ and its grain size increases during isothermal holding.

Mintz et al. [51] reported that DIF can be formed very readily in both fine and coarse austenite grained steels and much more rapidly than the ferrite from strain-free austenite. Very small strains ($\varepsilon=0.02$) are sufficient to induce the production of such ferrite, and the temperature range over which it appears span from just below the Ae3 temperature down to the undeformed $A_{e3}$ temperature. He reported that the prior austenite grain size has an important role on DIF. In coarse grained steels, deformation at low strain rates is concentrated along the grain faces, and the extensive DRV occurs, which is why the ferrite remains soft, so that only thin ferrite is able to form. At higher strain rates, work hardening takes place so that the strength of ferrite at high strains approaches that of the austenite. Under these conditions, the deformation is propagated towards the centres of the austenite grains and larger volume fractions of deformation induced the ferrite to form. In the fine-grained steels, the flow stress in the austenite grain boundary region is increased, so that when ferrite first forms, a considerable amount of work hardening takes place, which strengthens the ferrite. When combined with the increased number of triple points present in the materials, the increased work hardening promotes spreading of the deformation, with the results that larger volume fractions of ferrite are produced, even at low strains and strain rates.

Choi et al. [47] found that in order for the $\gamma$ to $\alpha$ transformation to occur during deformation, the strain must exceed a critical level, and further pointed out that during the deformation of austenite, DIF and DRX compete with each other, and one of them, which has the smaller critical strain, will take place ahead. When the deformation temperature is below Ae3, the critical strain for DIF is smaller than that for DRX, and DIF is the predominant softening mechanism, see Fig. 9. Because the prior austenite grain boundaries are the most potential nucleation site for DIF, the decrease in the prior austenite grain size accelerates the DIF kinetics.

Fig. 9. Schematic illustration of temperature dependencies of the critical strain for DIF (SIDT) and DRX [47].

Yang et al. [52] confirmed that the critical condition for DIF to occur in plain low-carbon steels is that DRX does not happen in the austenite during deformation, so that the energy of deformation can fully be used to induce the ferrite transformation.
Hurley and Hodgson [53] achieved a high level of grain refinement during this process in a steel with a large austenite grain size. They suggested that a high shear strain at the surface and a high undercooling are the main factors in the formation of UFF surface layers from very coarse austenite grains, and that the ferrite most likely forms by a process of rapid intragranular nucleation during, or immediately after, the deformation. They suggested that a bigger undercooling and a coarser prior austenite grain size are beneficial to supply a bigger undercooling. Priestner [54] further studied Hodgson’s results and confirmed that a critical nominal strain must be exceeded to trigger the near surface grain refining effect. Exceeding the critical strain increased the volume fraction of ferrite formed at the surface layer on cooling from the rolling temperature, while the core of the rolled plate continued to transform to a coarse microstructure of ferrite. However, thicker grain refined surface layers with a grain size of ~1 µm can be obtained after rolling the fine grained austenite with high rolling strains and fast cooling, in contrast to the contention of Hodgson and his co-workers [53], but in agreement with the conclusions of Mintz et al. [51], Yang et al. [52], and Choi et al. [47], etc.

DIF has been proven to be an effective route in refining the grain size of ferrite. Lower deformation temperature, microalloying elements and a finer prior austenite grain size are the factors affecting the grain size of ferrite. The factors affecting the ferrite grain size in this method will be discussed in more detail in Section 1.4.

1.3.7 Deformation in the dual-phase region

A steel with a grain size of 1.2 µm has been produced by heavy deformation in the α + γ dual-phase region [55, 56, 57]. A ferrite grain size of 3–4 µm can develop at 750°C, even if the prior austenite grains are very coarse, 300 µm [16]. Deformation in the dual-phase or in the ferrite regions appears to have the advantage that uniform elongation is improved together with increasing strength. Yield strength values in the range of 465-540 MPa and tensile strength in the range of 880-980 MPa, with the total elongation of 11-20%, have been reported for an 0.17C-0.44%Si-1.32Mn-0.15Nb-0.0013Ti steel [16].

1.3.8 Annealing of deformed pearlitic microstructure

It is reported that, when a pearlitic Fe-1.0C-1.4Cr steel has been cold rolled with a 70-95% reduction and annealed at 973 K, a grain size finer than 0.5 µm has been obtained [58]. Recently, in an European collaborative project, warm rolling of high-carbon steels with 0.6-1.2%C have been investigated and found to result in UFF with a spheroidised carbide aggregate structure [59].
1.3.9 Annealing of deformed martensitic microstructure

Recently Ueji et al. [60, 61, 62] have closely investigated the martensitic starting microstructure, cold-rolled at reductions of 25-75%, to obtain a UFF microstructure in a plain carbon steel (Fe-0.13% C) by annealing it at temperatures 500-600°C. A multiphase, ultrafine microstructure has been obtained composed of UFF grains (around 180 nm in size), uniformly precipitated nano cementite and tempered martensite, the fractions depending on the cold rolling reduction [62].

1.4 Influencing factors in the deformation-induced ferrite route

There are several processing parameters that can influence the final grain size of ferrite in the DIF route that were employed in the present study. Therefore, they are discussed in more detail here.

1.4.1 Strain

It has been confirmed that with increasing strain, more and finer DIF grains can be obtained. Up to now, many studies have been performed to clarify the dependence of DIF on the degree of deformation [47, 51, 52, 53, 54]. A relationship between the ferrite grain size and applied strain is shown in Fig. 10 [63]. A high strain is required, even though it is not so evident from Fig. 10, and it is one of the main difficulties in practice to supply.

![Fig. 10. Effect of strain on the grain size in the DIF route [63].](image)

As earlier mentioned, there exists a critical strain to form DIF [47, 51, 52, 55]. The critical strain is related with many factors, such as the deformation temperature, prior austenite...
As far as the critical strain is concerned, the strain rate is more important than the prior austenite grain size, contrary to the effect on the DIF transformed fraction. Among the processing parameters, the fine prior austenite grain size has the most pronounced influence on the promotion of DIF both by reducing the critical strain and highly accelerating the kinetics. The deformation temperature also has an important influence on the critical strain. Above $A_{c3}$, since ferrite is not thermodynamically stable, critical strain increases with a decreasing deformation temperature. However, in the regime below $A_{c3}$, the critical strain decreases with a decreasing deformation temperature. This relationship was shown in Fig. 9 [47].

The deformation modes are generally divided into two types, compressive and shear modes. Hodgson et al. [15] and Hurley[53] produced the UFF with only 30% deformation in the surface of a plate from coarse grained austenite, and one of the important reasons was thought to be the high shear strain at the surface. Inoue [64] studied the effect of shear deformation on grain refinement and concluded that the ferrite structures transformed from the deformed austenite are affected by not only the strain but the deformation mode, too. Hurley and Hodgson [65] studied the effect of pure shear strain on the efficiency of refinement in DIF, and found that it had a similar influence on the efficiency of DIF as compressive strain. Inoue et al. [66] further studied the different strain modes in multidirectional deformation and postulated that a concept of equivalent plastic strain can be used to estimate the efficiency of DIF.

**1.4.2 Strain rate**

It is generally thought that the rate of deformation has a small, or even negligible effect on refining the ferrite grain size during DIF. However, Mintz et al. [51] showed that the strain rate can have a certain influence on the fraction of DIF, especially as formed from the coarse austenite grains. If the strain rate is very low, then extensive DRV occurs and only thin ferrite film can form. However, if the strain rate is higher, then the work hardening takes place so that the strength of the ferrite at the high strains approaches that of the austenite. Under this condition, the deformation propagates towards the centres of the austenite grains and a larger volume fraction of deformation-induced ferrite is able to form. But for a finer austenite grain size, the strain rate has a lesser influence on the grain size and the fraction of DIF.

Yang et al. [52] investigated the effect of strain rate in plain low-carbon steels and found that the strain rate had some role on in the formation of DIF. For a given strain, a higher strain rate and a lower deformation temperature were beneficial for the formation of DIF. Hodgson and Hurley [65] studied the effects of processes variables on the formation of DIF in hot torsion and they found that the strain rate appeared to have some effect on the amount of ferrite formed.

Du et al. [67] suggested that the strain rate has an optimum range for DIF. The strain-induced transformation is a diffusional process so that increasing the strain rate has a negative effect on it. Decreasing the strain rate will increase the deformation time, and there would be enough time for the transformation to take place during the deformation.
Hence, there would be an upper limit for the strain rate at a given temperature. However, if the strain rate is very low, the strain-induced ferrite grains formed will have more time to grow.

1.4.3 Deformation temperature

Yada et al. [75] achieved a UFF of a fraction exceeding 50% by means of heavy deformation in the temperature region of $A_e - A_f$. Mintz et al. [51] reported that ferrite could be formed in the temperature regime of $A_e - (A_f - 20^\circ C)$. Yang et al. [52] obtained 90–95% ferrite of 2–3 µm grain size in a low-carbon steel by multipass deformation in the temperature range ($A_e + 30^\circ C$) – ($A_f - 20^\circ C$). Hodgson and coworkers [15, 53] obtained UFF in plain carbon steels by single-pass deformation just above $A_f$ and they suggested that this deformation temperature is very important for DIF. However, Priestner et al. [54] further confirmed that very fine DIF grains were also obtained at a deformation temperature up to 150°C above $A_f$. Bleck et al. [63] showed the positive effect of lowering deformation temperature on ferrite grain size at the strain of 1.2 for a C-Mn-Nb steel, as in Fig. 11a, but an even more pronounced effect is seen in Fig. 11b [68].

![Fig. 11. Effect of deformation temperature on grain size obtained in the DIF route (a) [63] (b) [68].](image)

DIF is stable between $A_e - A_f$, but unstable above $A_e$. If the holding time is long enough, DIF can transform back to austenite completely [52]. Deformation just above $A_f$ can supply the maximum undercooling for DIF, so $A_f$ is an important parameter. Hong et al. [69] have studied the effect of undercooling and they found that, when the strain is relatively small, increasing undercooling is very effective in increasing the amount of DIF, but at relative high strains, increasing the undercooling will affect only slightly the amount of DIF, but it can refine the DIF grain size.
1.4.4 Prior austenite grain size

The effect of the prior austenite grain size on DIF can be summarised as follows [51, 52, 54]:

1. A fine austenite grain can reduce the critical strain for DIF and therefore results in a finer ferrite grain size at a given strain.

2. The finer an austenite grain size, the higher the volume fraction of DIF at a given strain. Besides the enhancement of DIF, a smaller austenite grain size results in a more uniform ferrite microstructure after cooling. The size of retained austenite islands among the DIF grains is very important. The coarse austenite transforms (statically) to ferrite+pearlite or ferrite+bainite, whereas the fine austenite island transforms to fine ferrite and cementite particles.

3. A coarse austenite grain size can supply a higher undercooling by retarding the transformation of austenite to ferrite and therefore results in a finer grain size of DIF, at least under certain circumstances [15].

1.4.5 Steel chemistry

The microalloying elements such as Nb and Ti can have the following effects on DIF formation [70]:

1. Microalloying elements can reduce the critical strain for DIF by retarding the grain growth of austenite.

2. By widening the temperature range between non-recrystallization temperature and the Ar₃ temperature, a higher strain can be accumulated to exceed the critical strain for DIF, and a fine grain size can be more easily obtained.

3. Nb can make DIF to form at a higher start temperature of transformation and at a lower critical strain than in plain low-carbon steels, because of a higher strain accumulation rate in Nb-bearing steels that provides a larger driving force for the ferrite transformation and enhances the nucleation rate of ferrite.

Besides the microalloying elements, carbon content also has some important role on UFF formation. It has often been reported that UFF can be obtained not only in the microalloyed steels but also in plain carbon steels [1, 6, 7, 13, 14, 15, 16, 17, 18, 19]. Even in common C-Mn steels, UFF can be found in a multipass deformation with a short interpass time [14, 31]. Yada et al. [30] reported that they can obtain the UFF grain size (< 4 µm) in any steel with a carbon content from 0.02% to 0.3%C and Mn from 0.1% to 2% Mn without alloying elements or with the total alloy elements less than 3%. Hichson et al. [71] found that the chemical composition of the steel can slightly influence the morphology and volume fraction of UFF grains formed in the surface layers of a strip, and it significantly altered the microstructure formed in the core. In plain carbon grades, the level of ferrite refinement increased slightly, when the carbon content increased. However,
when the carbon concentration is below the solubility limit in the ferrite, finer and more DIF can be obtained that affects the mechanical properties beneficially [72], as shown in Fig. 12.

Fig. 12. The influence of carbon content on the mechanical properties of UFF steels [72].

1.4.6 Cooling rate

The effect of the cooling rate has an important role on DIF mainly by affecting the degree of undercooling [73]. Faster cooling will supply a bigger undercooling, which will decrease the critical strain for DIF and refine the grain size. However, the effect of the cooling rate has been found to decrease at above 10°C/s [74]. The cooling rate is believed to have a small effect on the volume fraction of DIF, but it acts as a controlling factor on the grain growth rate. Yada et al. [75] noticed that a cooling rate of 20°C/s is necessary to suppress the grain growth after hot working, and even a higher cooling rate is desirable.

It is worth mentioning here that an ultra fast cooling technology has been developed at CRM in Belgium [76]. Cooling rates 5–8 times higher than conventional ones in the temperature range of 400-650°C can be reached. With the aid of ultrafast cooling rates, the grain size of 2–3 µm in hot rolled strips has been attained.

1.5 Properties of ultrafine grained steels

As discussed earlier, when the grain size decreases, the yield strength and toughness are highly improved. For steels with 0.1-0.16%C, the maximum yield strength falls generally between 475–500 MPa, and the tensile strength is in the range 630-700 MPa, and the elongation about 13% for a grain size of 4 µm [77, 78]. With microalloying, the yield
strength can increase to 660 MPa and the tensile strength to 710 MPa with an elongation of about 18% [48, 78]. However, when the grain size of ferrite is refined to a 1 µm scale, the yield strength raises close to 800 MPa even in a C-Mn-Si steel [79] (see Fig. 1). However, some mechanical properties of UFF structure steels, the ductility in particular, are still of some concern (see Fig. 2).

1.5.1 **Strength and hardness**

The quantitative relationship proposed by Hall and Petch between the yield strength and the grain size in metals was given in Eq. (1). In Fig. 13, the proof/yield stress is shown as a function of the grain size for the bulk iron (Fig. 13a) [80] and for a low-carbon (0.15%C) steel (Fig. 13b) [81] revealing the Hall-Petch relation to be valid down to a very fine grain size. Clearly, a higher yield strength (up to 1600 MPa) can be achieved with an ultrafine grain size.

![Fig. 13. The relationships between the yield stress and the grain size, for iron (a) [80] and a low-carbon steel (b). For the low-carbon steel, the tensile strength is also shown [81].](image)

Shin et al. [82] obtained the relationship:

\[
YS (\text{MPa}) = 271 + 328 (\text{MPa} \, \mu m^{0.5}) \, d^{-0.5}
\]

for 0.15C-1.1Mn UFF steels with or without 0.06%V. The power of the grain size, i.e. the constant \(k_y\) in Eq. (1), is smaller for the UFF structure than generally reported for conventional steels. They suggested non-equilibrium low-angle grain boundaries to be the
reason for that. A recent relationship between the lower yield point (LYP) and the ferrite grain size in low-carbon steels is determined by Ohmori et al. [83] as:

\[
\text{LYP (MPa)} = 120 + 0.5 \times (\text{MPa m}^{0.5}) d^{-0.5}
\]  

(6)

This is close to the relationship given in Fig. 13a. The dependence of the yield and tensile strengths on the grain size is shown in Fig. 14 for conventional, fine and ultrafine (about 3 µm) grained 0.16C-0.20Si-0.80Mn steel strip [22]. A yield strength of around 600 MPa has been achieved with this refined grain size.

![Fig. 14. Dependence of the yield and tensile strengths on the grain size in conventional, fine and ultrafine (about 3 µm) grained 0.16C-0.20Si-0.80Mn steel strips [22].](image)

The relation between the tensile strength and the number of rolling cycles, resulting in a successive refinement of the grain size, is shown in Fig. 15 for an Ti-IF steel with a UFF structure processed by the ARB process (the grain size about 0.42 µm after 7 cycles) [81].

![Fig. 15. Mechanical properties of a Ti-IF steel after repeated cycles in the ARB process at 773 K [81].](image)
It has been testified that even when the grain is reduced to a 1 µm level, the hardness still obeys the Hall-Petch relationship, as shown in Fig. 16 [48]. The experimental relationship between the Vickers hardness (HV) and the ferrite grain size is:

\[ HV = 130 + 3.25D_{\alpha}^{-0.5} \]  

(7)

Fig. 16. Dependence of hardness on ferrite grain size for a Nb-bearing steel (a) and by powder-metallurgy processed iron and Fe-C alloy (b) [48, 80].

However, it appears that in the nanoscale range, there is an ambiguity in the applicability of the Hall-Petch relation. It seems that the limit for the validity of Eq. (1) is around a 0.2 µm grain size [84]. In agreement with this, the hardness for iron from the results of Takaki et al. [80] also reveals the validity of the Hall-Petch relation down to a grain size of about 0.2 µm.

1.5.2 Ductility

The stress-strain curve for an ultrafine grained steel was shown in Fig. 17, which that indicated quite a low ductility compared to that of a conventional C-Si-Mn steel. Some results of the tension tests are shown in Fig. 17 for an IF steel [85, 86] revealing clearly a decreasing elongation with decreasing grain size as well as low work hardening, especially when the grain size is below 1 µm. The relationship between the elongation and the grain size can also be seen in Fig. 18 and the fact seems to be that the uniform elongation becomes practically zero when the grain size is 1 µm or less [80, 87]. However, Howe [5] reports that the Corus UF-IF steel can give \( R_{p0.2} \approx 743 \) MPa, a tensile strength of 911 MPa with 19% total elongation.
Fig. 17. The typical stress-strain curves for (a) IF-steel ARB processed and annealed with different grain sizes [85], and Fe-0.13% C steel processed by the SRM route (b) 25% cold-rolling (d) 70% cold-rolling [62].

Fig. 18. The influence of grain size on elongation (a) for iron (b) for IF steel [80, 87].

Takaki et al. [80] showed the balance between uniform elongation and the yield strength for different microstructures (Fig. 19). According to them, in a C-Mn steel with a 0.93 µm ferrite-carbide structure, a uniform elongation of about 10% can be achieved with a 600 MPa yield strength, a combination equivalent to that of the quench and tempered martensitic steels. The plain ferrite with a 1.7 µm grain size and nil-ductility is a steel of the 1970’s from Morrison and Miller, see [87].
Fig. 19. The balance between the uniform elongation and the yield strength for different microstructures in a C-Mn steel [80].

It is found that the reduction in area values of UFF grained steels are generally somewhat larger than those of coarse grained alloys [36, 88]. The UFF grained alloys, in which the abrupt yielding occurs are followed by Luder’s strain, which may exhibit plastic instability, particularly at low temperatures and at high strain rates. At temperatures above about 0.5 of the melting temperature, UFF grained alloys become superplastic, having very large elongations. This is a consequence of the high strain-rate sensitivity of the flow stress. [36, 88].

Recently, it has been proposed that a bimodal microstructure, UFF with some coarser grains, would enhance ductility without too much scarifying the strength [89, 90, 91, 92]. Mughrabi et al. [91] suggested that a low temperature annealing resulting in recovery instead of recrystallization would be a route to obtain a particularly recovered ultrafine grain size or a bimodal grain structure for optimal properties. Jin and Lloyd [92] processed an ultrafine grained Al-3.1Mg-0.3Mn alloy by asymmetric rolling and annealing and introduced 20-45% coarser grains (about 4 µm) into the microstructure. They measured a tensile elongation of 16% at 22% fraction of coarser grains among ultrafine grains compared to 8% with a 100% ultrafine (average about 1 µm) structure. Gill Sevillano and Aldazabal [90] has discussed the chances of ductilization.

Howe [93] reports that at CRM an ultrafine (about 2 µm) dual-phase μ-strip (0.07C-1.4Mn-0.03Nb) has been processed, using cold-rolling and annealing steps, with a 490 MPa yield strength, 860 MPa tensile strength and 10% uniform elongation.

1.5.3 Work hardening rate

The ability for strain hardening must be present to prevent the premature failure due to the force instability. The problem of ultrafine or nanocrystalline ductile materials is not
brittleness but the fact that their work hardening rate is very low, even negative, as evident from Fig. 17, which may cause problems in actual applications. It appears that achieving the desired strength levels and ductility would not be a big problem but the lack of a work hardening rate is a field which needs to be concerned more [32]. Recently, it has been suggested that a grain size distribution with some fraction of grains large enough to sustain dislocation activity has been shown to be effective in providing the needed strain hardening and therefore increased ductility. Second phase particles in nanocrystalline matrices should also be a pathway for optimization of strength and ductility by providing strain hardening [93]. Recently Ohmori et al. [83] investigated the strain-hardening behaviour of UFF low-carbon steels with dispersed cementite particles and showed that strain-hardening and consequently uniform ductility can be improved with an increasing volume fraction of cementite particles, as a result of increasing density of geometrically necessary dislocations generated by local non-uniform plastic deformation.

1.5.4 Toughness

Heslop and Petch [95] have also shown that the ductile to brittle transition temperature is inversely proportional to \( d^{-1/2} \). When a polygonal ferrite grain structure is obtained by a deformation in the austenite recrystallization or in the non-recrystallization region, the transition temperature is given by:

\[
T_{rs} = \alpha + \beta d^{-1/2}
\]  

(8)

where \( \alpha \) is a constant depending on chemical composition, and \( \beta \) is a constant representing the degree of resistance of grain boundaries to the brittle crack propagation. Takaki et al. [80] presented the relationship as

\[
DBTT (K) = 330 - 0.33 d^{-0.5}
\]  

(9)

for steels with conventional or UFF grain size.

Not many reports are found on the toughness of ultrafine grained steels, partly because the samples in which the UFF grain structure has been obtained are quite small and insufficient for actual testing. However, with surface fine-grained plates with a grain size of 2 µm in a 25 mm plate, the Charpy-V transition temperature was -196°C [29]. In a 0.15%C-Mn-Si steel with a grain size less than 1 µm, the ductile-brittle transition temperature was lower than 77 K [96], i.e. the toughness seems to be excellent.

Recently, Hanamura et al. [97] have tested the impact toughness of several microstructures, ultrafine ferrite-cementite among others. The average grain size of the UFF-cementite steel (0.15%C-0.33%Mn-Si) was 2.7 µm (>5° misorientation). Fig. 20a shows the Charpy-V transition curves revealing a low transition temperature about 120 K for the UFF-cementite structure. Nagai [87] also reports that the Charpy-V toughness of the UFF grained 0.15C-0.39Si-1.43Mn steel with cementite in the
microstructure is clearly better than for the ferritic-pearlitic structure, as shown in Fig. 20b.

Fig. 20. The absorbed impact energy as a function of test temperature for ferrite/pearlite (F/P), ultrafine ferrite/cementite (Uf/C), quenched (Q), and quench and tempered (QT) steels (a) [97] and for various grain sizes (b) [87].

Fig. 21 shows the relationship between the effective grain size and DBTT for various steel microstructures. For the UFF structure, the DBTT is extremely low, about 50 K [97]. However, the upper self energy decreases down to 50 J when the grain size decreases to 1 μm.
Concerning the fracture toughness properties of UFF structures, it has been reported that the crack arrest toughness was about 6 kN/mm\(^{1.5}\) in the surface layer of a 25 mm plate (Nippon Steel: SUF) having a 2 µm grain size [29].

1.5.5 Cyclic response

There is only a limited number of studies on the fatigue behavior of ultrafine grained materials. They have, however, revealed a cyclic softening behavior occurring in UFF steels, as shown in Fig. 22 [33]. The flow stress remains much lower in cyclic loading than the monotonous tensile stress-strain curve (Fig. 22a). This kind of cyclic instability is similarly as in high-strength QT-steels, as seen in Fig. 22b. Morimoto et al. [93] observed the bending fatigue strength/tensile strength ratio to be 0.52 for an ultrafine grained (3-5 µm) steel sheet.
Fig. 22. The behavior of UFF structure in fatigue loading. Tensile and cyclic stress-strain curves indicating cyclic softening (a), the position of UFF steels compared with some other microstructures (b) [33].

It has been observed that the initial crack growth rate is somewhat higher in a UFF steel than in a conventional coarse-grained steel as seen in Fig. 23 [33]. Also, the threshold value of $\Delta K$ is slightly higher. However, at longer cracks the crack growth rate becomes lower than in conventional steel.

Fig. 23. Fatigue crack propagation rates in a low carbon (0.15C) steel in as-received (30 µm grain size) or with UFF structure (0.5 µm) conditions [33].
1.6 Thermal stability of ultrafine grained steels in fabrication processes

Microstructure should be sufficiently stable over a useful period of time to retain its properties. Standard, root-time grain growth equations are frequently expressed in the form [98]:

\[
\frac{1}{d^n - d_0^n} = Ct
\]  

(10)

where \(d_0\) is the average grain diameter before the growth, \(n\) is an exponent close to 0.5 and \(C\) is a material parameter depending on the mobility (highly temperature dependent) and the specific energy of a grain boundary [98]. However, the normal grain growth is not the only growth mechanism and it is often superseded by abnormal growth.

An ultrafine grain size represents a very high grain boundary area, and thus a high interfacial stored energy. The stored energy as a driving pressure for growth is typically on the order of 0.5 MPa for micrometer-sized subgrains [98]. Therefore, it can be assumed that a very rapid grain growth would be exhibited by the ultrafine grained materials. However, Xu et al. [99] observed that in an Al-Zn-Mg-Zr alloy, a grain size of 0.3 \(\mu\)m was very stable at elevated temperatures, up to 400°C (1 hour) due to fine Al\(_2\)Zr precipitates. Hodgson et al. [26] noted that micrometer-size grains in steels were stable even after 30 min at 700°C. They ascribed this to very heavy pinning exerted by the cementite particles on the transformed ferrite grain boundaries (conventional pearlite was not being observed after their SIDT processing route). Shin et al. [100] reported that ultrafine grained ferrite/pearlite microstructures were stable in annealing at temperatures up to 510°C for one hour. Bhadeshia [101] claims that very fine grains are self-pinning, assuming that the boundary curvature is required for the energetically driven coarsening to occur, and very short boundary lengths will inhibit this. There is some support for this from Shvindlerman [102]. Driver [98] discussed the stability of nanocrystalline materials and proposed that the recovery process can transform a highly deformed microstructure into a relatively low-energy configuration. Annealing to extended recovery can develop a well-defined subgrain structure, which is quite equiaxed and it may be relatively stable due to a spatially homogeneous distribution of boundary curvatures and energies.

Shin et al. [100] observed that the activation energy of grain growth for a UFF structure, processed by ECAP and only recovered, was about 106 kJ/mol, which value is lower than the activation energy for volume diffusion in ferrite (280 kJ/mol), for the grain boundary diffusion (164 kJ/mol) or for the grain boundary mobility of pure iron (147 kJ/mol), i.e. about 35% of that for self-diffusion of Fe in the ferrite. Hence, the low-angle grain boundaries created by intense deformation show higher mobility compared to ordinary boundaries. After recrystallization, the activation energy of grain growth was markedly higher, about 230 kJ/mol.

In welding, thermal cycles are exerted by the heat introduced and grain coarsening can occur in steels and consequently some softening. There is some doubt concerning the weldability of UFF steels. They are lean in chemical composition and thus, inherently well
weldable, but the UFF grain size would have to be maintained in the weldment, and its heat affected zone (HAZ). There are indications that the thermal stability of UFF grains depends on the route by which they are obtained [89], but the weld thermal cycle would exceed any stability criteria.

Fig. 24 shows the hardness distribution across the HAZ for three ultrafine grained steel plates, 12 mm in thickness, as CO₂ welded at 42 kJ/cm (260 A, 33V, 17 cm/min). The base metal hardness is 230 HV (Steel C and D) or 260 HV (Steel E) and the minimum hardness values were around 150 HV at the HAZ at temperatures around Ac₃ [103]. It was observed that no grain coarsening took place below Ac₁ and the maximum grain size in the intercritical zone was 2.7-3.8 µm. However, at the fusion line, the austenite grain size was about 170-190 µm and the hardness 220 HV (steel with 0.1%C, CEV 0.35). A small amount of Nb and Ti are believed to prevent further the grain growth. At a heat input of 9 kJ/cm (t₈/₅ =5 s), the softened zone was found to be 2 mm in width, and at 40 kJ/cm (t₈/₅ =27 s), 6 mm in width [104]. Using the 500 MPa class filler wire, the hardness of the weld metal was higher than 240 HV, i.e. a tensile strength of about 800 MPa [104,105].

Similar results were obtained, as shown in Fig. 25, for another three ultrafine grained steels with a strength of 780 MPa, 16 mm in thickness, in MAG welding at 10 kJ/cm (328A, 28.5 V, 54 cm/min) and 20 kJ/cm (328 A, 31.5 V, 32 cm/min). The results indicated that softening occurred in the HAZ because of the coarsening of ferrite grains due to welding heat input [106]. However, by using a low welding heat input and a strength-overmatching weld metal, the detrimental effect of softening on the strength was restrained, so that the welded joints had a strength equivalent to that of the base metal we obtained. The ultra-narrow gap welding method is being proposed for welding UFF grained plates [107].

![Fig. 24. Hardness distribution across the HAZ of three ultrafine grained steels [103].](image)
41

Fig. 25. Hardness distribution across three welded joints in steels of D (0.14%C-0.30%Si-
1.46%Mn), E (0.095%C-0.30Si-1.45%Mn) and F (0.093%C-0.30Si-1.45Mn-0.016%Nb-
0.007%Ti), MAG welded at different heat inputs (a) 10 kJ/cm (b) 20 kJ/cm [106].

1.7 The scope of the work

As described in the previous Sections 1.3-1.6, extensive research has been undertaken and
is going on all over the world to develop UFF steels. However, before commercializing the
processing, still further research is required, especially on those processes that are
potentially transferable to hot rolling mills, similarly as for on the mechanical properties
achieved and on the thermal stability of the fine structure in workshop fabrication
operations. Therefore, in the present study, the objectives put forward have been first to
investigate the chances to manufacture ultrafine grained steels by some methods, in
principle convenient to mass-production, and secondly to test the thermal stability of
ultrafine grained microstructures. Testing of mechanical properties remained out of the
scope of this study. The content of the work can be listed as the following:

(1) Clarify the importance of the processing variables affecting the microstructure
obtained by severe plastic deformation at a low austenite regime in C-Mn and microal-
loyed steels.
(2) Systematically investigate the influence of the initial microstructure, including ferrite, pearlite, bainite, martensite and tempered martensite, on the final ferrite grain size in the processing route consisting of cold rolling and subsequent recrystallization annealing.

(3) To explain the effect of steel chemical composition, especially the role of second phase particles, cementite or microalloy carbides, on the ferrite grain size refinement.

(4) To investigate the thermal stability of UFF structure in annealing treatments.

(5) To investigate briefly the stability of UFF microstructure in low-heat input joining, such as electron and laser beam welding.

Due to extremely intensive global academic and industrial interest and activity in this challenging field, a huge number of papers have been published in journals and presented in conferences over five recent years [59, 108, 109, 110, 111, 112, 113, 114]. This is true especially in Japan, where two national conferences are being held annually. Most of papers published are in Japanese and they have not been referred here. Annually, The International Symposium on Ultrafine Grained Structures, ISUGS, has been held, the latest in Shanghai in April, 2004 [113]. Also in the USA the topic has been active in connection with TMS annual conferences [109, 110]. However, most of the publications are quite brief and concentrated on restricted matters compared to the present work which is more comprehensive, offering a more general understanding on the formation of UFF structures in certain industrially-potential processes and the controlling factors in them. Especially, the chemical composition range covered in the present study, the carbon content from 0.006 to 0.8 mass-\%, is much wider than generally before, and particularly, the refining mechanism in various deformed microstructures and the influences of the precipitate particles and the processing route on the thermal stability of the UFF microstructure have been investigated and discussed here for the first time to this broad extent. Some results have been published in a conference paper [114] and also printed in a journal [115].
2 Experimental procedures

2.1 Materials, sampling and deformation

In the first part of the experiments, the DIF processing route was applied to four low-carbon steels, C-Mn, Nb-bearing, Nb-Ti and a high Ti-bearing, coded as Nb-hTi, steel. These common steels were selected to investigate the effect of microalloying on the formation of UFF grain structure and on thermal stability. In the second part, the static recrystallization of various deformed microstructures (SRF/SRM route) was investigated, where, in addition to the above mentioned four steels, also four high-carbon (0.28-0.80% C) steels and one ultralow-carbon (ULC) bainitic steel were used. All steels except ULC, were commercial grades (the first four delivered by Rautaruukki Oyj and the steels 0.3-0.6C by Imatra Steel Oyj companies). The steel 0.8C was obtained from a Chinese company. The chemical compositions of all the materials used in this work are listed in Table 1.

Table 1. Chemical compositions of the steels (mass-%).

<table>
<thead>
<tr>
<th>Steel grade</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>Cu</th>
<th>Cr</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Mn</td>
<td>0.09</td>
<td>0.32</td>
<td>1.01</td>
<td>0.012</td>
<td>0.036</td>
<td>0.002</td>
<td>0.003</td>
<td>0.28</td>
<td>0.50</td>
<td>0.0042</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>0.15</td>
<td>0.30</td>
<td>1.42</td>
<td>0.012</td>
<td>0.002</td>
<td>0.037</td>
<td>0.033</td>
<td>0.01</td>
<td>0.02</td>
<td>0.0070</td>
<td></td>
</tr>
<tr>
<td>Nb-Ti</td>
<td>0.07</td>
<td>0.21</td>
<td>0.96</td>
<td>0.009</td>
<td>0.001</td>
<td>0.028</td>
<td>0.046</td>
<td>0.11</td>
<td>0.0070</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb-hTi</td>
<td>0.07</td>
<td>0.17</td>
<td>1.50</td>
<td>0.015</td>
<td>0.001</td>
<td>0.029</td>
<td>0.048</td>
<td>0.13</td>
<td>0.0080</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.3C</td>
<td>0.28</td>
<td>0.26</td>
<td>1.18</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.4C</td>
<td>0.39</td>
<td>0.28</td>
<td>1.21</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.6C</td>
<td>0.60</td>
<td>0.30</td>
<td>1.00</td>
<td>0.025</td>
<td>0.025</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.8C</td>
<td>0.80</td>
<td>0.2</td>
<td>0.80</td>
<td>0.030</td>
<td>0.030</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ULC</td>
<td>0.006</td>
<td>0.20</td>
<td>1.65</td>
<td>0.020</td>
<td>0.07</td>
<td></td>
<td></td>
<td>0.01</td>
<td>0.10</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The low-C and ULC steels: Quantovac analyses by Rautaruukki Oyj. 0.3-0.8C steels: nominal compositions from the brochures.
The DIF tests were performed on a Gleeble-1500 thermomechanical simulator at the Materials Engineering Laboratory. Graphite foils were used as lubricant in order to obtain a more uniform distribution of strain in axisymmetric compression of cylindrical specimens.

Pieces of the C-Mn and Nb steels were cut from the 25 mm thick rolled plates, austenitizing at 1150°C for 30 min in an air furnace, water quenched and subsequently annealed at 605°C for 60 min. In the case of the Nb-Ti and Nb-hTi steels, the specimens were cut from 30 mm thick plates, reheated at 1250°C for 30 min, water quenched and subsequently annealed at 605°C for 30 min, before they were heated in the Gleeble simulator at 20°C/s to the certain reheating temperature for a given time, cooled at 2°C/s to the given deformation temperature and held for 5 s before the compression to a given strain and finally cooled at different cooling rates between 1-20°C/s. In some experiments, cut pieces of C-Mn, Nb, Nb-Ti and Nb-hTi steels were first cold-rolled in a laboratory rolling mill to certain strains before cylindrical specimens were machined (ø 10 x 12 mm) and tested in the Gleeble. If not mentioned, the specimens were not applied to the cold-rolling stage. In certain experiments, repeated reheating cycles were employed to refine the austenite grain size.

In the static recrystallization of deformed martensite (SRM) route, all specimens were cut into strips with a length of 150~250 mm, a width of 30~50 mm, and a thickness of 10~25 mm. For the C-Mn and Nb steels, the pieces were reheated in a furnace at 1000°C and 1100°C, respectively, for 1 h and water quenched. The Nb-Ti and Nb-hTi steel pieces were reheated at 1250°C for 2 h and water quenched. The steels 0.3C, 0.4C and 0.6C were reheated at 900°C for 30 min and water quenched and subsequently tempered at 450, 500 and 550°C for 30 min, respectively. Cold-rolling of the martensitic steel pieces was performed in an one-stand laboratory rolling mill in multi-pass deformation to the total reduction, in most tests 70% (i.e. the true strain 1.2), and subsequently annealed at different temperatures for various times. In the static recrystallization of deformed pearlite (SRP) route, the specimens of the 0.8C steel were cut from heavily drawn (1.6 strain) pearlitic wire with a diameter of 5 mm to pieces with the length of 5 mm, and subsequently annealed at different temperatures for various times.

2.2 Dilatometry, metallography and hardness testing

The start temperature of the austenite decomposition A_{r3} was determined at various cooling rates by dilatometric measurements using the Gleeble 1500. Cylinders similar to those in the DIF tests were used.

An optical microscope (OM) and a scanning electron microscope (SEM, Jeol JSM-6400 at the Institute of Electron Optics) were employed to investigate the microstructures. A solution of 1-5% HNO₃ + 95% ethyl alcohol was used to etch the metallographic specimens for ferritic microstructures. In a few examinations, a SEM equipped with an EBSD (Link Opal) device was also used to determine the grain size distribution and the misorientation of grain boundaries. At the time of these examinations, the measurements took quite a long time due to a low sampling rate of the EBSD equipment so that this
useful technique was not utilized to the extent it could be. At present, the system is much faster with the new camera and software.

Different methods were employed to determine the ferrite grain size depending on the grain morphology. In the case of equiaxed grains, the linear intercept method was adopted, and in the case of pancaked grains, the circle method was used. To account for the nonhomogeneous distribution of the grain size in the cross-section of the specimen, the OM or SEM photos were taken from various positions around the centre of the specimen. If the grain size was measured with the SEM at a magnification above 5000, then at least five SEM photos were taken from different fields in each sample. The volume fraction of DIF grains were determined with the ASTM point counting technique using the 5 x 5 grid.

The hardness was measured by a Vickers pyramid method with a 10 kg load. To guarantee randomness, at least 3 positions were measured around the centre section. In welded seams, the hardness distributions were measured in cross-sections across the welds.

### 2.3 Thermal stability tests

Thermal stability tests were performed in annealing at a temperature of 600°C or above in an air furnace or in a salt bath by repeatedly reheating the specimens for given durations. The microstructures were checked at certain intervals.

In welding tests, the thermal stability of the UFF structure in the C-Mn, Nb-Ti and Nb-hTi steel strips, obtained by the SRM route, were investigated. The tests were performed at Lappeenranta University of Technology, Department of Mechanical Engineering. The electron and laser beam welding methods were used autogeneously without a filler metal. The test parameters are listed in Tables 2 and 3. In principle, lower and higher heat inputs were applied.

*Table 2. The parameters in the electron beam welding tests for the C-Mn, Nb-Ti and Nb-hTi steels.*

<table>
<thead>
<tr>
<th>Grade</th>
<th>Sheet thickness, mm</th>
<th>Voltage, kV</th>
<th>Current, mA</th>
<th>Welding speed, cm/min</th>
<th>Heat input, J/cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Mn</td>
<td>1.1</td>
<td>150</td>
<td>2.6</td>
<td>100</td>
<td>234</td>
</tr>
<tr>
<td>Nb-Ti</td>
<td>1.3</td>
<td>150</td>
<td>2.3</td>
<td>100</td>
<td>207</td>
</tr>
<tr>
<td>Nb-hTi</td>
<td>2.4</td>
<td>150</td>
<td>4.5</td>
<td>100</td>
<td>405</td>
</tr>
<tr>
<td></td>
<td>2.4</td>
<td>150</td>
<td>5.0</td>
<td>100</td>
<td>450</td>
</tr>
</tbody>
</table>
Table 3. The parameters in the laser welding tests for the C-Mn, Nb-Ti and Nb-hTi steels.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Sheet thickness, mm</th>
<th>Power, kW</th>
<th>Welding speed, cm/min</th>
<th>Heat input, J/cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Mn</td>
<td>1.1</td>
<td>4.8</td>
<td>650</td>
<td>443</td>
</tr>
<tr>
<td></td>
<td>1.1</td>
<td>2.0</td>
<td>100</td>
<td>1200</td>
</tr>
<tr>
<td>Nb-Ti</td>
<td>1.3</td>
<td>4.8</td>
<td>650</td>
<td>443</td>
</tr>
<tr>
<td></td>
<td>1.3</td>
<td>2.0</td>
<td>100</td>
<td>1200</td>
</tr>
<tr>
<td>Nb-hTi</td>
<td>2.4</td>
<td>4.8</td>
<td>420</td>
<td>686</td>
</tr>
<tr>
<td></td>
<td>2.4</td>
<td>2.5</td>
<td>100</td>
<td>1500</td>
</tr>
</tbody>
</table>
3 Results

3.1 $A_{\text{r3}}$ temperatures

In the DIF route, the idea is to perform heavy deformation at or just above the $A_{\text{r3}}$ temperature of the steel that is dependent on the chemical composition, the degree of deformation and the cooling rate. Therefore, those temperatures were determined before the DIF experiments by the dilatometric tests. As an example, typical dilatometric curves are plotted for the C-Mn steel in Fig. 26 just to demonstrate the technique. The effects of the degree of deformation (none or 0.8 strain) and the cooling rate are shown. The influence of deformation seems to be quite small, but the cooling rate affects the transformation temperatures more significantly. From the curves, it can be concluded that for the deformed austenite, the $A_{\text{r3}}$ temperature at the cooling rate of 10°C/s is about 755°C.

Fig. 26. Typical dilatometric curves at various cooling rates for C-Mn steel without deformation (a) and as deformed at 0.8 strain at 850°C (b).
Values of the measured $A_{\text{r}3}$ temperatures for the C-Mn, Nb, Nb-Ti and Nb-hTi steels are listed in Table 4.

Table 4. $A_{\text{r}3}$ temperatures of the steels.

<table>
<thead>
<tr>
<th>Steel grade</th>
<th>Route</th>
<th>$A_{\text{r}3}$, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Mn</td>
<td>Reheated to 900°C for 2 min, deformed to 0.9 strain and cooled at 20°C/s</td>
<td>760</td>
</tr>
<tr>
<td>Nb</td>
<td>Reheated to 950°C for 2 min, deformed to 0.9 strain and cooled at 10°C/s</td>
<td>775</td>
</tr>
<tr>
<td>Nb-Ti</td>
<td>Reheated to 1000°C for 2 min, deformed to 0.9 strain and cooled at 10°C/s</td>
<td>780</td>
</tr>
<tr>
<td>Nb-hTi</td>
<td>Reheated to 1000°C for 2 min, deformed to 0.9 strain and cooled at 10°C/s</td>
<td>780</td>
</tr>
</tbody>
</table>

It can be noticed that for the low-carbon steels used here, the differences in $A_{\text{r}3}$ are quite small and therefore more or less the same deformation temperatures could be used in the DIF tests, 780°C in most tests.

3.2 DIF structures

3.2.1 DIF structure in the C-Mn steel

The initial austenite grain size was determined by reheating and then cooling at 2°C/s to the deformation temperature (but without deformation) and quenching into water. A martensitic structure obtained by quenching from 780°C after reheating to 900°C for 2 min is shown in Fig. 27. From the figure, it is seen that the prior austenite grain structure was somewhat nonuniform, but the mean grain size was fine, about 11 µm.
Fig. 27. Prior austenite grain structure of the C-Mn steel as reheated to 900°C for 2 min, water quenched from 780°C.

The applied strain forms ferrite from the austenite as-strain induced during the compression. However, a fully strain-induced transformed structure is hard to get in the DIF route. Fractions of DIF grains as deformed at 780°C with strains of 0.6, 0.9, and 1.2, were found to be about 46, 53 and 67%, respectively. Fig. 28 shows the dual-phase ferrite-martensite microstructure, as deformed at 0.6 and quenched. In quenching immediately after the deformation, the remaining untransformed austenite has transformed into martensite (a darker phase). The ferrite grain size is about 5.5 µm.

Fig. 28. Microstructure of the C-Mn steel with a DIF fraction of 46%, as reheated to 900°C for 2 min, deformed at 0.6 at 780°C and water quenched.

A typical microstructure as reheated to 900°C for 2 min, deformed at 780°C with a strain of 0.9 and cooled at a rate of 20°C/s is shown in Fig. 29. The microstructure consists of ferrite (white), martensite (ash). The ferrite grain size is about 4.0 µm, and the martensite grains are generally somewhat larger.
The grain sizes obtained at different deformation temperatures, strains and cooling rates in the DIF route are summarized in Table 5. As seen, even increasing the strain up to 1.2, the UFF grain size was not achieved, but only grain sizes of around 3-4.5 µm.

Table 5. The influence of deformation conditions on the ferrite grain size and hardness of the C-M steel in the DIF route.

<table>
<thead>
<tr>
<th>Reheating temperature /time</th>
<th>Deformation temperature, °C</th>
<th>Strain</th>
<th>Cooling rate, °C/s</th>
<th>Ferrite grain size, µm</th>
<th>Hardness, HV</th>
</tr>
</thead>
<tbody>
<tr>
<td>900°C, 2 min</td>
<td>760</td>
<td>0.9</td>
<td>1</td>
<td>4.3</td>
<td>165</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>760</td>
<td>0.9</td>
<td>10</td>
<td>4.1</td>
<td>182</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>760</td>
<td>0.9</td>
<td>20</td>
<td>4.0</td>
<td>194</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>760</td>
<td>0.9</td>
<td>WQ</td>
<td>242</td>
<td></td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>780</td>
<td>0.9</td>
<td>1</td>
<td>4.2</td>
<td>176</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>780</td>
<td>0.9</td>
<td>10</td>
<td>4.1</td>
<td>181</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>780</td>
<td>0.9</td>
<td>20</td>
<td>4.0</td>
<td>192</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>780</td>
<td>1.2</td>
<td>10</td>
<td>3.6</td>
<td>177</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>780</td>
<td>1.2</td>
<td>20</td>
<td>3.2</td>
<td>195</td>
</tr>
<tr>
<td>900°C, 2 min</td>
<td>800</td>
<td>0.9</td>
<td>20</td>
<td>4.3</td>
<td>176</td>
</tr>
<tr>
<td>850°C, 2 min</td>
<td>780</td>
<td>0.9</td>
<td>20</td>
<td>4.0</td>
<td>185</td>
</tr>
<tr>
<td>950°C, 30 s</td>
<td>780</td>
<td>0.9</td>
<td>20</td>
<td>4.0</td>
<td>187</td>
</tr>
</tbody>
</table>
Besides the deformation temperature, strain and the cooling rate, the influences of the strain rate in the range of 0.1 to 20 s\(^{-1}\) were investigated, as deformed at 780°C with a strain of 0.9, but no difference could be found in the microstructure or in the grain size.

Because the grain size remained quite coarse, several ways to refine the prior austenite grain size were tried. Using a lower reheating temperature of 850°C instead of 900°C (strain 0.9 at 780°C) a ferrite grain size of 4.0 µm was achieved, i.e. it was practically unchanged, see Table 5. However, a finer ferrite grain size of 2.7 µm was obtained starting from a martensitic microstructure that was reheated to 850°C only for 1 min, and then deformed with a strain of 1.2 at 780°C. This microstructure is shown in Fig. 30.

![Fig. 30. Microstructure in the C-Mn steel obtained from the initially martensitic structure as reheated to 850°C for 1 min, and compressed at 1.2 strain at 780°C. The grain size of the ferrite is 2.7 µm.](image)

The prior austenite grain size was refined to 8 µm, when a tempered (600°C, 40 min) martensitic specimen was cold rolled at a strain of 1.2 and then reheated to 900°C for 2 min. However, the ferrite grain size was as coarse as 3.2 µm when deformed at 780°C with a strain of 1.2, as shown in Fig. 31. The second (black) phase seems to be a kind of carbide aggregate presumably due to nonhomogeneous distribution of carbon in the tempered martensite and short reheating cycle that has not dissolved the coarse carbides.
Fig. 31. Microstructure from a cold-rolled tempered martensitic specimen, reheated at 900°C for 2 min and compressed at 1.2 strain at 780°C and cooled at 20°C/s. The grain size of ferrite is 3.2 µm.

Besides a lower reheating temperature or cold rolling, a prior austenite grain size can also be refined by the phase transformation from ferrite to austenite. Using the double reheating cycle, first reheating at 880°C for 1 min, followed by cooling at 20°C/s to 200°C without any deformation, then again heating to 880°C for 1 min, and deformation at 780°C with a strain of 1.2, the ferrite grain size was as fine as 2.0 µm, as shown in Fig. 32.

Fig. 32. Microstructure of C-Mn steel as deformed at 780°C with a strain of 1.2 after refining the prior austenite grain size by the repeated ferrite-austenite transformation cycle. The ferrite grain size is about 2.0 µm.

If the C-Mn specimen was cold-rolled at the reduction of 70% (the strain of 1.2) before the double reheating cycle, even a finer ferrite grain size of 1.8 µm was obtained, as shown in Fig. 33.
Fig. 33. Microstructure of the C-Mn steel as deformed at 780°C with a strain of 1.2 after refining the prior austenite grain size by cold-rolling (strain of 1.2) and repeated reheating cycle. The grain size of the ferrite is 1.8 µm.

Still further, starting with a martensitic microstructure deformed at a strain of 0.7 and a double reheating cycle before the deformation at 780°C with a strain of 1.2, the grain size of ferrite could be refined to about 1.3 µm, as seen in Fig. 34.

Fig. 34. Microstructure of the C-Mn steel as deformed at 780°C at the strain of 1.2 after a double reheating cycle on the initial deformed martensite. The grain size of the ferrite is about 1.3 µm.

The results demonstrate the vital importance of a fine initial austenite grain size to obtain a UFF grain structure at a reasonable reduction.
3.2.2 DIF structure in the Nb-steel

The measured value of $A_{r3}$ of the Nb-steel was around 775°C as reheated to 950°C for 1 min and cooled at the rate of 20°C/s. Therefore, a deformation temperature of 775°C was used.

By quenching the specimen after the deformation, it could be noticed that different DIF fractions, 26%, 44% and 60%, were obtained at the strains of 0.3, 0.6, and 0.9 as reheated to 900°C for 2 min and deformed at 775°C, as shown in Fig. 35 a, b and c, respectively. The hardness values were 345, 297 and 266 HV, respectively. The martensite (prior austenite) regions seem to be quite large.

![Fig. 35. Microstructure in the Nb-steel with different DIF fractions (the darker phase) after deformation at 775°C at (a) strain 0.3, fraction 26% (b) strain 0.6, fraction 44% (c) strain 0.9, fraction 60%.

The ferrite grain sizes of the Nb steel at different deformation temperatures, strains and cooling rates in the DIF route are summarized in Table 6. It can be noticed that the smallest grain size is 3.3 µm, i.e. a real UFF grain size could not be reached by this way (Fig. 36). Nevertheless, the grains formed from the deformed retained austenite seem to be relatively fine without forming any contiguous regions.

Table 6. The influence of deformation conditions on the ferrite grain size of a Nb steel.

<table>
<thead>
<tr>
<th>Reheating temperature/time</th>
<th>Deformation temperature, °C</th>
<th>Strain</th>
<th>Cooling rate, °C/s</th>
<th>Ferrite grain size, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>950°C, 1 min</td>
<td>900</td>
<td>0.6</td>
<td>10</td>
<td>5.3</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>900</td>
<td>0.9</td>
<td>10</td>
<td>5.1</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>850</td>
<td>0.6</td>
<td>10</td>
<td>5.1</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>850</td>
<td>0.9</td>
<td>10</td>
<td>4.1</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>775</td>
<td>0.6</td>
<td>10</td>
<td>4.5</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>775</td>
<td>0.9</td>
<td>5</td>
<td>3.6</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>775</td>
<td>0.9</td>
<td>10</td>
<td>3.4</td>
</tr>
<tr>
<td>950°C, 1 min</td>
<td>775</td>
<td>0.9</td>
<td>20</td>
<td>3.3</td>
</tr>
<tr>
<td>900 °C, 2 min</td>
<td>775</td>
<td>0.6</td>
<td>10</td>
<td>4.5</td>
</tr>
<tr>
<td>900 °C, 2 min</td>
<td>775</td>
<td>0.9</td>
<td>10</td>
<td>3.4</td>
</tr>
<tr>
<td>900 °C, 2 min</td>
<td>775</td>
<td>1.2</td>
<td>10</td>
<td>3.3</td>
</tr>
</tbody>
</table>
A typical microstructure, as reheated to 900°C for 2 min, deformed at 775°C with a strain of 1.2 and cooled at the rate of 20°C/s, is shown in Fig. 36.

Fig. 36. Microstructure in the Nb-steel as reheated to 900°C for 2 min, deformed with the strain of 1.2, cooled at the rate of 10°C/s. The ferrite grain size is 3.3 µm.

Similarly as in the case of C-Mn steel, special schedules were employed to refine more the prior austenite grain size. Then, with the specimen cold-rolled to a strain of 1.2 before reheating to 900°C for 2 min, deforming at 775°C with the strain of 1.2 and cooling at a rate of 10°C/s, a ferrite grain size of 2.1 µm was obtained, as shown in Fig. 37.

Fig. 37. Microstructure in the Nb steel as a result of the route: cold-rolling to the strain of 1.2, reheating to 900°C for 2 min, deforming at 775°C with a strain of 1.2 and cooling at a rate of 10°C/s. The ferrite grain size is 2.1 µm.

In the double reheating route, as described in Section 3.2.1, heated twice to 880°C for 2 min and subsequently deformed at 775°C at a strain of 1.2 and finally cooled at a rate of 10°C/s, a ferrite grain size of 1.7 µm was obtained, as shown in Fig. 38.
Fig. 38. Microstructure in the Nb steel as deformed at 775°C with a strain of 1.2 following the double 880°C for a 2 min reheating cycle. The ferrite grain size is 1.7 µm.

In the specimen, cold-rolled at a the strain of 1.2 and then twice reheated and deformed at 775°C with a strain of 1.2, the grain size of ferrite was further refined to 1.4 µm, see Fig. 39.

Fig. 39. Microstructure of Nb steel after the route: cold-rolling at 1.2 strain, reheating twice at 880°C for 2 min, deformed at 775°C with a strain of 1.2. The ferrite grain size is 1.4 µm.

3.2.3 DIF structure in Nb-Ti steel

Processed according to the conventional TMCP route, reheating at 1200°C for 2 min, deformed at the temperatures of 950, 900 and 850°C at a strain of 0.9 and cooled at the rate of 20°C/s, the ferrite grain sizes obtained in the Nb-Ti steel were 9.5, 5.6 and 4.9 µm.
respectively. At a higher strain of 1.2 at 850°C and cooling at 10°C/s, the ferrite grain size became somewhat smaller, 3.5 µm.

Lowering the deformation temperature to 780°C and 750°C and using a lower reheating temperature of 1000°C, a strain of 1.2 and a cooling rate of 10°C/s, the ferrite grain size was refined to 2.8 µm, as seen in Fig. 40. However, the microstructure, when deformed at 750°C, consisted of coarse elongated proeutectoid ferrite grains, revealing that the deformation had occurred below Ar3. This was in accordance with a measured Ar3 temperature of 780°C (Table 4).

![Fig. 40](image1.png)

**Fig. 40.** Microstructure in Nb-Ti-steel as deformed at 780°C with a strain of 1.2 after reheating at 1000°C for 2 min. The ferrite grain size is 2.8 µm.

Applying a complex processing route, with cold-rolling at a strain of 1.2, reheating at 900°C for 2 min, and deformation at 850°C with a strain of 0.7, the ferrite grain size obtained was not finer than 3.4 µm. However, when deformed at 780°C at a strain of 1.2, the ferrite grain size was quite fine, 1.1 µm, as shown Fig. 41.

![Fig. 41](image2.png)

**Fig. 41.** Microstructure in Nb-Ti-steel as cold-rolled at 1.2, reheated at 900°C for 2 min, deformed at 780°C with a strain of 1.2 and cooled at 20°C/s. The ferrite grain size is 1.1 µm.
Using a double reheating cycle, 880°C for 2 min, cooling at 20°C/s to room temperature and heating back to 780°C for 2 min, then deforming at a strain of 1.2 and cooling at 10°C/s, the ferrite grain size remained quite coarse, 2.0 µm, as shown in Fig. 42.

Fig. 42. Microstructure in Nb-Ti steel as deformed at 780°C at a strain of 1.2 following a double reheating (880/780°C, 2 min) cycle. The ferrite grain size is 2.0 µm.

3.2.4 DIF structure in the Nb-Ti steel

In specimens of the Nb-Ti steel, when reheated to 1200°C for 2 min and deformed with strains of 0.6, 0.9 and 1.2 at 800°C and cooled at the rate of 10°C/s, the ferrite grain sizes were about 6.0, 4.6 and 3.5 µm, respectively, i.e. they remained above the UFF grain sizes.

Using a lower reheating temperature of 1000°C for 2 min and strains of 0.6, 0.9 and 1.2 at 780°C, the ferrite grain sizes were somewhat refined, 3.8, 2.9 and 2.4 µm, respectively. As an example, the microstructure obtained at the strain of 1.2 is shown in Fig. 43.
Fig. 43. Microstructure in Nb-hTi steel as reheated to 1000°C for 2 min and deformed at 780°C with a strain of 1.2. The ferrite grain size is 2.4 µm.

Using the cold-rolled specimen and the same reheating procedure, a strain of 1.2 at 780°C resulted in a much finer ferrite grain size, about 1.1 µm, as seen in Fig. 44.

Fig. 44. Microstructure in Nb-hTi steel obtained from a cold-rolled specimen as reheated at 1000°C for 2 min and deformed at 780°C at the strain of 1.2. The ferrite grain size is 1.1 µm.

Employing a double reheating cycle, first reheating to 880°C for 2 min, cooling at 20°C/s to room temperature and reheating back to 780°C for 2 min, then deforming at a strain of 1.2 and cooling at 10°C/s, the ferrite grain size of 2.1 µm was obtained, as shown in Fig. 45. It seems that elongated grains are present among the equiaxed ones in the microstructure.
Fig. 45. Microstructure in Nb-hTi steel as deformed at 780°C with a strain of 1.2 after a double reheating cycle. The ferrite grain size is 2.1 µm.

Hence, a grain size as fine as 1.1 µm could be obtained in Nb-hTi steel, similarly as in Nb-Ti steel, but only if relatively complex treatments to pre-refine the austenite grain size were utilized. Heavy cold rolling before a low-temperature reheating seems to be the most effective method for this.

3.3 Annealing of various deformed initial microstructures

In the second stage, heavy cold-deformation was applied to various steels with different initial microstructures, which were subsequently isothermally annealed to recrystallize the structure in order to obtain a UFF ferritic grain size. However, it was realized in preliminary tests that the grain size achieved was not very fine in all cases, even with special pre-treatments performed. However, high-carbon microstructures provided UFF grain size more easily.

3.3.1 Annealing of deformed ferrite

With C-Mn steel, the special procedure employed was as follows: cold rolling at a the strain of 1.2, reheating to 900°C, hot rolling at 850°C with a total reduction of 50% in two passes and air cooling, then cold rolling at a the strain of 0.8 and annealing at 600°C for various times. A ferrite grain size of 3.6 µm in a fully recrystallized microstructure was obtained in this route.
With Nb steel, cold-rolling at a strain of 1.2 and annealing at different temperatures with various times were tried. As a result, the ferrite grain size was 4.6 µm in a fully recrystallized microstructure, as annealed at 640°C for 40 min.

With Nb-Ti steel, the following procedure was employed: cold rolling at a strain of 1.2, reheating to 900°C for 30 min, hot rolling at 850°C with a total reduction of 50% in two passes, air cooling, cold rolling at a strain of 0.9 and annealing at 600°C for 20 min, a ferrite grain size of 3.2 µm was obtained, as shown in Fig. 46.

![Fig. 46. Microstructure in Nb-Ti steel refined by cold rolling (strain 1.2), hot rolling (50% reduction in two passes) before the second cold rolling (strain 0.9) and annealing at 600°C for 20 min. Ferrite grain size is 3.2 µm.](image)

In Nb-hTi steel, with the first cold rolling at a strain of about 0.8, hot rolling (i.e. reheated to 900°C for 30 min, deformed at 850°C with a total reduction of 50% in two passes, followed by air cooling), and cold rolling at a strain of 0.9 and annealing at 600°C for 20 min, a ferrite grain size of 2.4 µm was obtained. A typical microstructure is shown in Fig. 47.

![Fig. 47. Microstructure in Nb-hTi steel refined by cold rolling (strain 1.2), hot rolling (50% reduction in two passes), before the second cold rolling (strain 0.9) and annealing at 600°C for 20 min. Ferrite grain size is 2.4 µm.](image)
Hence, it can be concluded that annealing of cold-rolled low-carbon ferritic structures and the static recrystallization of them does not result in a very fine ferritic grain size.

3.3.2 Annealing of deformed pearlite

As observed in the previous section, annealing of cold-rolled ferrite in the low-carbon C-Mn or microalloyed steels did not result in a very fine grained structure. Therefore, steels with other initial microstructures and/or high-carbon content were tested to reveal whether they have an effect on the formation of UFF structure.

In experiments with a fully pearlitic initial structure, a high-carbon steel of 0.8C was used as a test material. The initial microstructure is shown in Fig. 48.

![Fig. 48. The initial pearlitic microstructure of 0.8C steel.](image)

Cold-drawing the steel wire at a strain of 1.6 and annealing at 700°C for 1 hour, a fully recrystallized microstructure with a ferrite grain size of about 0.75 µm was obtained. In addition to the ferritic matrix, the microstructure contains lot of cementite particles, with a coarse diameter of 0.5 µm, always located at the grain corners, as shown in Fig. 49.
3.3.3 Annealing of deformed bainite

A high-carbon steel of 0.6C was used to investigate grain refinement in a bainitic microstructure. The bainitic microstructure, shown in Fig. 50, was obtained on the Gleeble in the route: reheating to 900°C for 2 min, cooling at 40°C/s to 350°C and holding for 5 min. This steel was deformed at the strain of 1.2 at 700°C and subsequently annealed at the same temperature for various times until a fully recrystallized microstructure was obtained.
A fully recrystallized microstructure with a very fine ferrite grain size of 0.8 µm, and the diameter of cementite particles of about 0.3 µm, was obtained in annealing at 700°C for 50 min, as shown in Fig. 51.

Fig. 51. Microstructure in 0.6C steel obtained from the bainite as deformed at a strain of 1.2 and annealed at 700°C for 50 min. The mean grain size of ferrite is 0.8 µm.

3.3.4 Annealing of deformed martensite

A martensitic initial microstructure was also tried instead of a ferritic one, with a route termed here as SRM. The C-Mn and Nb steels were reheated at 1000°C and 1100°C, respectively, for 60 min and water quenched to get the martensitic structure. Also, the Nb-Ti and Nb-hTi steels were reheated at 1250°C for 2 h and water quenched. Cold-rolling of the martensitic steel pieces was performed in a laboratory rolling mill in multi-pass deformation to a total reduction of 70% (strain 1.2) in the case of the C-Mn and Nb steels, and to a reduction of 90% for Nb-Ti and Nb-hTi steels. Cold-rolled pieces were subsequently annealed at different temperatures for various times until a fully recrystallized microstructure was obtained. Typical structures are displayed in Figs. 52–58. It can be realized that relatively fine grain sizes of 1-1.5 µm were obtained in the Nb and C-Mn steels and therefore this route might be a relevant one when considering the industrial processing of UFF steels.
Fig. 52. Microstructure in C-Mn steel when annealed at 600°C for 1 h from deformed (strain 1.2) martensite. The mean grain size of ferrite is 1.5 µm.

Fig. 53. Microstructure in Nb steel when annealed at 700°C for 10 min from deformed martensite. The mean grain size of ferrite is 1 µm.
Fig. 54. Microstructure in Nb-Ti steel when annealed at 700°C for 15 min from deformed martensite. The mean grain size of ferrite is 2.4 µm.

Fig. 55. Microstructure in Nb-hTi steel as annealed at 700°C for 90 min from deformed martensite. The grain size of ferrite is 2.6 µm.

A specimen of C-Mn steel, reheated at 1000°C 2 min and quenched to get martensite, was deformed to a 60% reduction (strain of 0.9) and annealed at 600°C for 60 min. In this case, the EBSD technique was used to analyse the grain structure. The orientation image and the grain size distributions are shown in Fig. 56a and b respectively, based on grain boundary misorientation ≥15°. According to this figure, the average grain size is 1.79 µm (ASTM 16.8) and the average equal circle diameter is 1.51 µm. These values are in agreement with the result shown in Fig. 52, that a grain size of 1.5 µm obtained at a strain of 1.2. Hence, a real UFF grain size was achieved. If a grain boundary misorientation ≥5° was selected, the average grain size value was even smaller, 1.37 µm (ASTM 17.2). This indicates that there are quite many low-angle grain boundaries in the structure as shown in Fig. 56c and d. In addition, it can be seen from Fig. 56b that there are a few larger grains of up to 9 µm...
present in the microstructure. This is just to demonstrate that the EBSD technique would certainly provide lot of useful information on the microstructure, especially on the homogeneity and regarding the distinction between cells and grains, but at the time of the study, the technique was quite slow, so that it was not utilised to any broader extent. It can, however, be highly recommended for future studies.

Fig. 56. EBSD results for C-Mn steel, as quenched from 1000°C into water, deformed with a 0.9 strain at 780°C and annealed at 600°C for 1 hour. (a) grain orientation image (b) grain size distribution (c) grain boundary misorientations indicated by the colours (red >15°, green > 5°) (d) misorientation distribution.

It is interesting to notice that a distinctly a coarser grain size was obtained in the Nb-Ti and Nb-hTi steels than in the C-Mn and Nb steels. This seems to be the fact, even considering that the annealing treatments were specific for each steel, and determined to result in a recrystallized structure without grain growth. Hardness was also used to follow the progress of softening.
3.3.5 Annealing of deformed tempered martensite

High-carbon steels of 0.3C, 0.4C and 0.6C were reheated at 900°C for 50 min, water quenched to get martensite and then tempered at 500°C for 1 h. Subsequently, the steels of 0.3C and 0.4C were cold-rolled at a the strain of about 1.2 and the 0.6C steel at a strain of 0.9. Finally, the specimens were annealed at 600°C for various times. As a result, the ferrite grain size smaller than 1 µm was successfully obtained in all the steels, as shown in Figs. 57~59. In addition to ferrite, numerous cementite particles are present in all steels, with an the estimated diameter of about 0.3 µm.

Fig. 57. Microstructure of 0.3C steel as annealed at 600°C for 60 min from deformed tempered martensite. The grain size of ferrite is 0.9 µm.

Fig. 58. Microstructure of 0.4C steel when annealed at 600°C for 60 min from deformed tempered martensite. The grain size of ferite is 0.7 µm.
A conclusion can be drawn that from high-carbon steels, whatever their initial microstructure, pearlite, bainite, martensite or tempered-martensite, a microstructure of UFF-carbide can be obtained by cold-deformation and subsequent annealing to recrystallize the deformed structure.

3.4 Thermal stability of ultrafine grained steels in annealing

The thermal stability of the ultrafine grained structure in steels has an important influence on potential applications. Due to the large total grain boundary area of the UFF grained structure, the stored energy is high and therefore the tendency to coarsening of the grain size in service at elevated temperatures, heat-assisted bending operation or in welding, can be expected to be pronounced. Here, the thermal stability of UFF grained structures obtained by both the DIF and SRM routes was tested.

3.4.1 C-Mn steel

*Microstructure from the DIF route:* Earlier in Section 3.4, it was shown that a grain size of about 2.0 µm could be obtained in the DIF route by utilizing a repeated reheating cycle to refine the prior austenite grain size and deforming at 780°C with a strain of 1.2 (see Fig. 32). Some secondary phase was present among ferrite, obviously carbide aggregate or pearlite. This UFF material was used in the annealing tests.

It was observed that during isothermal annealing at a temperature of 700°C, the UFF grain size remained almost constant until an annealing time of 170 min, as shown in Fig.
60. Then, quite suddenly, abnormal grain growth appeared. The pearlite phase became spheroidised during the soaking.

Fig. 60. Microstructure in C-Mn steel in DIF specimens as annealed at 700°C for (a) 150 min (b) 170 min.

**Microstructure from the SRM route:** Microstructure in the C-Mn steel from the SRM route, as annealed at 600°C for 1 h from the deformed (strain 1.2) martensite, was ferrite with a mean grain size 1.5 µm (see Fig. 52). Contrary to the behavior of the microstructure obtained by the DIF route, the ferrite grains in the SRM specimens exhibited normal grain growth during the isothermal annealing at 600°C, and the grain sizes were about 2.5, 3.1 and 5.5 µm after holding times of 10, 40 and 90 min, respectively, as shown in Fig. 61.
Fig. 61. Microstructures in C-Mn steel in SRM specimens as annealed at 600°C for (a) 10 min (b) 40 min (c) 90 min.
3.4.2 Nb-Ti steel

Microstructure from the DIF route: It was earlier shown that by applying a complex processing route, cold-rolling at a strain of 1.2, reheating at 900°C for 2 min, and deforming at 780°C at a strain of 1.2, the resulting ferrite grain size was quite fine, 1.1 µm (see Fig. 41). Some pearlite or carbide aggregate was also present in addition to the ferrite. This UFF material was used in annealing tests.

Similarly to the behavior of the C-Mn steel in the DIF specimens, during isothermal annealing at 600°C, no grain coarsening was observed (Fig. 62a) and also at 700°C the ferrite grain size remained practically constant until a soaking time of 180 min, when abnormal grain growth appeared, as seen Fig. 62 (b-d). The minor secondary phase became totally spheroidised during this period.

Microstructure from the SRM route: The SRM route resulted in a fully recrystallized microstructure (at 700°C for 15 min) with a mean ferrite grain size of 2.4 µm and the carbide particles were distributed uniformly (see Fig. 54). This microstructure was soaked at 600°C. Then, similarly as in the C-Mn steel, the ferrite grains exhibited normal grain growth to the sizes of 2.5, 3.2, 3.9 and 5.5 µm within the annealing time of 10, 30, 60 and 90 min, respectively, as shown in Fig. 63.

Fig. 62. Microstructures in Nb-Ti steel in DIF specimens as annealed at (a) 600°C 820 min (b) 700°C 150 min (c and d) 700°C 180 min.
Fig. 63. Microstructures in Nb-Ti steel in a SRM specimen in the course of annealing at 600°C for (a) 10 min (b) 30 min (c) 60 min (d) 90 min.

3.4.3 Nb-hTi steel

Microstructure from the DIF route: The microstructure in the Nb-hTi steel, obtained from the DIF route consisting of cold-rolling, reheating at 1000°C for 2 min and deformation at 780°C at a strain of 1.2, was ferrite with the UFF grain size of 1.1 µm (see Fig. 44). Similar to the behavior of the DIF specimens of the C-Mn and Nb-Ti steels, during annealing at 700°C, the ferrite grain size remained constant until a soaking time of 240 min, when a abnormal grain growth started, as shown in Fig. 64.

It has to be noticed that, although the grain growth in all of the DIF specimens started by the same abnormal mode, the incubation time was somewhat different, 170, 180 and 240 min in the C-Mn, Nb-Ti and Nb-hTi steels, respectively. Hence, it tends to increase with an increasing alloying content of the steel.
Fig. 64. The grain growth in Nb-hTi steel in a DIF specimen during isothermal annealing at 700°C for (a) 150 min (b) 240 min (c) 300 min.

**Microstructure from the SRM route:** The microstructure in the Nb-hTi steel from the SRM route, annealed at 700°C for 90 min from the deformed martensite, was ferrite with a grain size of 2.6 µm with carbide particles distributed uniformly on the grain boundaries.
as well as at grain interiors (see Fig. 55). A specimen was annealed at 700°C. Similarly as the behaviour of the C-Mn and Nb-Ti SRM specimens, the ferrite grain size grew continuously to 3.8, 4.3, 5.0 and 6.5 µm within the soaking times of 60, 120, 150 and 210 min, respectively, as illustrated in Fig. 65.

![Fig. 65. The grain growth in Nb-hTi steel in a SRM specimen during isothermal annealing at 700°C for (a) 60 min (b) 120 min (c) 150 min (d) 210 min.](image)

### 3.4.4 High-carbon steels

In order to reveal the influence of the carbon content, some SRM (tempered martensite) specimens of the 0.3C and 0.4C steels with very fine grain sizes were selected to investigate the thermal stability of UFF microstructures at 600°C and 700°C. It was observed that in the 0.3C steel, the abnormal grain growth seemed to start within 110 min at 600°C, as seen in Fig. 66. The microstructure of the 0.4C steel remained unchanged within this time, but at 700°C, abnormal grain growth was found in 2.5 hours, as shown in Fig. 67.
3.5 Thermal stability in electron and laser beam welding

3.5.1 Hardness distribution

Thin sheets (about 1 mm in thickness) of C-Mn, Nb-Ti and Nb-hTi steels, processed by the SRM route as described in Section 3.4, were welded autogeneously by the electron and laser beam methods to get a rough idea about the stability of the UFF grain structure in the course of welding. Two inputs, relatively low and high were applied. The hardness distribution in the welded joints were measured and typical results are shown in Figs. 68–70. In all the welded seams, both in electron and laser beam welded and high and low heat input welds, the hardness increased from the base metal towards the HAZ and the weld metal, and no softening was found.
Fig. 68. Hardness distribution in a C-Mn steel electron beam welded seam (the heat input of 234 J/cm).

Fig. 69. Hardness distribution in a Nb-Ti steel electron beam welded seam (the heat input of 207 J/cm).
3.5.2 Microstructures in welded seams

**C-Mn steel:** The microstructure of C-Mn steel base metal is shown in Fig. 71, as seen in an optical microscope and SEM. The UFF grain size was about 1.5 µm.

![Fig. 71. Base metal microstructure of C-Mn steel seen in (a) OM and (b) SEM.](image)

Three macrographs of the seams are shown in Fig. 72. One typical HAZ microstructure close to the fusion line and one microstructure of the base metal-HAZ region are shown in Figs. 73 and 74, respectively. No coarse grained zones were found.
Fig. 72. Seams in C-Mn steel (a) electron beam welding at a heat input of 234 J/cm (b) laser welding at a heat input of 443 J/cm (c) laser welding at a heat input of 1200 J/cm.
Fig. 73. Microstructure of C-Mn steel across the fusion line in a seam electron beam welded with a heat input of 234 J/cm.

Fig. 74. Microstructure of C-Mn steel between the HAZ (left) and the base metal (right) in a seam laser welded with a heat input of 1200 J/cm.
**Nb-Ti steel:** The base metal microstructure of steel Nb-Ti is shown in Fig. 75. The seams welded by the electron beam and laser methods are shown in Fig. 76 (a), (b) and (c), respectively. One typical HAZ microstructure across the fusion line, and one typical microstructure between HAZ and the base metal are shown in Figs. 77 and 78, respectively.

![Base metal microstructure of Nb-Ti steel.](image)

Fig. 75. Base metal microstructure of Nb-Ti steel.
Fig. 76. Electron beam welded joint in Nb-Ti steel (a) electron beam welded seam (b) laser welded seam at a heat input of 443 J/cm (c) laser welded seam at a heat input of 1200 J/cm.
Figs. 77 and 78. HAZ microstructure in Nb-Ti steel in electron and laser beam welded seams.

*Fig. 77. HAZ microstructure in Nb-Ti steel in an electron beam welded seam.*

*Fig. 78. HAZ microstructure in Nb-Ti steel in a laser welded seam at a heat input of 1200 J/cm².*

**Nb-hTi steel:** The typical microstructure of Nb-hTi base metal is shown in Fig. 79. The seams welded by the electron and laser beam methods are shown in Figs. 80 and 81, respectively. A typical HAZ microstructure across fusion line and a typical microstructure between HAZ and base metal are shown in Figs. 82 and 83, respectively.
Fig. 79. Microstructure of Nb-hTi base metal.
Fig. 80. Seams in Nb-hTi steel electron beam welded at a heat input (a) 405 J/cm (b) 450 J/cm.
Fig. 81. Seams in Nb-hTi steel, laser welded at heat input (a) 686 J/cm (b) 1500 kJ/cm.
Fig. 82. HAZ microstructure in Nb-hTi steel across the fusion line in a seam electron beam welded at a heat input of 450 J/cm.

Fig. 83. Microstructure in Nb-hTi steel between the HAZ and the base metal in a seam laser welded at a heat input of 1500 J/cm.
4 Discussion

4.1 Refinement of the ferrite grain size from deformed single-phase austenite or ferrite

The DIF route, the dynamic transformation of ferrite from heavily strained fine-grained austenite, was confirmed to be a method to obtain a UFF microstructure. The method has been presented earlier in the literature, as described in the Introduction. In the present work, the factors influencing on the grain size achieved were extensively investigated in four low-carbon steels, C-Mn and Nb, Nb-Ti and Nb-hTi microalloyed steels. The experimental results, in agreement with the previous ones, e.g. Priestner, Bleck [54, 63], showed that the controlling parameters in the DIF route are: refined prior austenite grain size, low deformation temperature and high strain. According to the present results, a specially pre-refined prior austenite grain size was quite crucial to reach the UFF grain size (≤ 3 µm) level. Unfortunately complicated for practical purposes, special schedules for cold rolling of the martensitic structure or repeated reheating cycles had to be employed to refine the austenite grain size to the extent that the UFF grain size could be obtained.

A strain rate in the range of 0.1~10 1/s was not found to affect significantly the grain size, in agreement with the results of Du et al. [67], who proposed an optimum deformation rate for the formation of DIF.

It is well known that the cooling rate can also play an important role on the final grain size of ferrite in the TMCP route, such as increasing the undercooling, as indicated by Eqs. (3) and (4), but in the DIF route, the influence of the cooling rate on the grain size is not significant. For instance, in the C-Mn steel, at the cooling rates of 1, 10 and 20°C/s as deformed at 780°C with a strain of 0.9, as listed in Table 5, the final ferrite grain size only varied slightly. Similar results were obtained for the Nb steel, as listed in Table 6. However, the cooling rate can play a more important role regarding the type of the second phase present. Because in the C-Mn steel, for instance, even with the strain of 0.9, the volume fraction of DIF is only about 53%, which means that 47% of deformed austenite, enriched in carbon will transform into different phases depending on the cooling rate. It
seemed that in practice, some kind of carbide aggregate was present at low cooling rates and martensite at high ones. The hardness of the final microstructure increased from 176 to 192 HV, when the cooling rate increased from 1 to 20°C/s revealing the martensite substituting the carbide aggregates.

In fact, it can be proposed that the final desirable structure would consist of two phases, such as UFF and martensite, i.e. to have “an improved dual-phase steel” structure that might presumably possess a better ductility and better work hardening characteristics than those in the plain UFF structure (see Fig. 2). As mentioned in the Introduction, at CRM an ultrafine (about 2 µm) dual-phase strip (0.07C-1.4Mn-0.03Nb) has been processed with a 490 MPa yield strength, 860 MPa tensile strength and 10% uniform elongation [5]. At Manchester Materials Science Centre, a PhD work will start (in Autumn 2004) on ultrafine grained two-phase steels [117], for they seem to offer considerable advantages in terms of fully exploiting the property advantages of UFF grained steels. In another way, a bimodal grain structure, which can improve ductility, may be obtained by partial recrystallization of severely deformed metal or by the control of its deformation/annealing cycle, as discussed by Gil Sevillano and Aldazabal [90].

The DIF route has an obvious disadvantage when a high strain is required. Yada et al. [75] reported that a DIF volume fraction of 50% required a reduction of 80%. Hodgson et al. [26] used the reduction of 35–40%, but then only a very thin surface layer of UFF was obtained. Further, the efficiency of multipass deformation has been proved to be lower than that of single-pass deformation [30]. Here, without special pre-refinement of the austenite grain size, even at a strain of 1.2 at 780°C, the ferrite grain sizes remained as 3.6, 3.0, 2.8 and 2.4 µm in the C-Mn, Nb, Nb-Ti and Nb-hTi steels, respectively, i.e. they were fine but not very fine.

As long as the deformation is performed close to Ar3, a low deformation temperature cannot contribute any more to the refinement of the ferrite grain size. Therefore, the only way to obtain further refinement or to reduce the requirement of severe deformation is to refine the prior austenite grain size. In Japan Sumimoto Metal Industries has already in 1975 developed a modification of TMCP, called SHT (Sumimoto High Toughness) involving rough rolling above Ar3, cooling below Ar1 and reheating back just above Ar3 to get fine grained austenite before the finishing rolling below the non-recrystallization temperature of austenite [116]. Leinonen [118] has similarly proposed that a UFF grain size can be obtained at a reasonable reduction using an extra low-temperature reheating stage following the pre-rolling and before the rolling below the non-recrystallization temperature. In the present work, this was investigated in numerous experiments, where in addition to a low reheating temperature, the prior austenite grain size was refined by cold rolling or using double reheating cycles. A reduced reheating temperature and a short soaking time only decrease the austenite grain growth, but it cannot further refine the austenite grain size. With the aid of cold rolling or repeated reheating cycles, a UFF microstructure was obtained at a strain of 1.2, i.e. at a rolling reduction of 70%, as shown by the results summarised in Table 7. Heavy cold rolling of ferrite, or preferably martensite, before a low-temperature reheating seems to be the most efficient method when seeking for the finest grain sizes.
The results also indicate that with an increasing microalloying content somewhat finer grain sizes were obtained, i.e. a higher refinement efficiency (defined here as the easiness, a kind of efforts necessary, to obtain a UFF grain size). The reason for that could be related to the retarded austenite grain growth during a low-temperature soaking, resulting from the Ti-alloying or the higher conversion ratio of ferrite/austenite due to undissolved Ti-bearing particles.

The effect of carbon on the refinement efficiency in the DIF route was not studied in the present work. Hodgson et al. [15] found that the carbon content can only have a slight effect on that. However, Liu et al. [72] reported that ultralow carbon (0.003%C) content can enhance refinement efficiency, for the whole deformation energy can be used for the strain-induced transformation, because nothing is needed for the diffusion of carbon atoms.

In principle, there are two methods to refine the recrystallized ferrite grain size: a severe deformation or refining the initial ferrite grain size by a low-temperature reheating.
cold rolling and DIF processing before the deformation and annealing. The static recrystallization of ferrite (SRF) route was found to be relatively ineffective to refine the ferrite grain size, as the results in Section 3.3 showed. It was difficult to obtain UFF in any of the low-carbon steels of C-Mn, Nb-Ti and Nb-hTi from the ferrite phase. This indicates that the carbide particles in the low-carbon ferrite cannot exert any important influence on the refinement in the SRF route. Later it will be shown that the carbides, however, have an important role on the thermal stability of the UFF structure in preventing grain growth. Hence, the number of nucleation sites in recrystallization may remain relatively low in deformed ferrite, compared to those in pearlite, bainite or martensite, as discussed later.

It can be concluded that UFF grain structure can be obtained from deformed austenite by the DIF route or a grain size of 2.4-4 \( \mu \)m from single phase ferrite by the SRF route, but both routes are highly dependent on severe deformation and the initial grain size. Concerning the efficiency, the DIF route is preferable, but for economical manufacturing, the treatments to refine the prior austenite grain sizes are quite complex.

4.2 Refining mechanisms in annealing of other deformed microstructures

4.2.1 Refining mechanism in annealing of deformed pearlite

A pearlitic structure can be obtained by a cooling of the austenite over a wide range of carbon concentrations. It is composed of blocks of lamellar colonies, i.e. a prior austenite grain is divided into several blocks and an individual block is divided into a few colonies with differently inclined ferrite-cementite lamellae. Inside a block, ferrite orientation is identical. Hence, a pearlitic structure is characterised by the lamellar spacing, colony size and block size [119-124].

The ferrite phase is plastically much softer than cementite. During plastic deformation, pearlite behaves heterogeneously at several microstructural levels. The heterogeneous plastic deformation occurs not only between ferrite and cementite, but also between blocks with different orientations. Heterogeneous plastic deformation may also be induced between lamellae with different inclinations and different lamellar spacings within an individual block [119-124].

Plastic straining in pearlite mainly takes place in the ferrite phase and only to a small extent in cementite. However, it has been found that plastic straining can also occur in cementite so that the thickness of lamellae decreases with the increasing strain [124]. Recent studies on the microstructure of heavily cold-rolled pearlite have revealed that cementite lamellae break into nanocrystals and they are forced to dissolve into ferrite during deformation [120].

The strain in cold-rolled ferrite of the pearlite phase is inhomogeneous. With increasing strain, the microstructure consists of greatly elongated slab-like cells separated by
boundaries and the misorientation between the cells increases and the cell dimensions decrease.

Recently, a UFF structure has been reported to be obtained by annealing the deformed pearlitic microstructure, but the mechanism has not been given[58]. In a European project, ultrafine ferrite-spheroidised carbide structures have been processed by warm rolling in high carbon steels (0.6-1.2%C) [59]. Here, in 0.8C steel with pearlitic microstructure deformed with a strain of 1.6, a fully recrystallized microstructure with a ferrite grain size of 0.8 mm and fine cementite particles (the diameter about 0.2-0.5 mm) was also obtained, as was shown in Fig. 49. This means that during the annealing, not only the deformed ferrite recrystallized, but also the deformed cementite lamellae spheroidised rapidly, aided by a high diffusion rate due to dislocations and vacancies created by plastic deformation. It could be thought that grain size is refined in deformed pearlite similarly as in the static recrystallization of single-phase ferrite. However, it was difficult to obtain UFF grains in the SRF route, as the results in Section 3.3 showed. There it was concluded that the number of nucleation sites in the deformed ferrite are low in low-carbon steels. Therefore, obviously in the route of annealing of deformed pearlite there is another, more effective mechanism than that acting in deformed ferrite. This higher refinement efficiency must be related to pearlite microstructure. In principle, this can be a result of two factors: a large number of nucleation sites and/or the retardation effect by the cementite particles on the recrystallized grain growth.

However, deformation is essential in pearlite, as found also here experimentally by annealing the undeformed pearlitic microstructure of 0.8C steel. Then, only a grain size larger than 20 µm was obtained, as shown in Fig. 84. This experiment revealed that only a few effective nucleation sites exist in the pearlitic microstructure without deformation.

Fig. 84. Microstructure of the 0.8C steel after annealing at 700°C for 6 hours.

It is well known that recovery and recrystallization are strongly affected by the precipitation state. Fine dispersoids can retard both processes, but large particles (>1 µm), on the other hand, are generally assumed to promote recrystallization by the particle stimulated nucleation mechanism (PSN) [125]. PSN in the recrystallization has been found in many alloys, including those of aluminum, iron, copper and nickel [125, 126, 127]. The main parameters, which determine whether or not PSN occurs, are the strain and
the particle size, a higher strain and a bigger particle size enhancing PSN. There is also an evidence that nucleation occurs preferentially at prior groups of particles [127]. The plateform particles with a size of 200 x 10 nm have been found to be able to stimulate a nucleation in a steel after a heavy deformation of 90% [128]. During deformation of pearlitic microstructure, the cementite lamellae break into pieces, whose size can be expected to be bigger than the above-mentioned 200 x 10 nm that can stimulate the nucleation (the diameter of cementite particles was estimated as 0.2-0.5 µm). Therefore, presumably the spheroidised cementite lamellae play a role by the PSN mechanism supplying a large number of nucleation sites for recrystallization, thereby resulting in a high refinement efficiency. Mizoguchi et al. [58] studied in detail the microstructure of deformed pearlite by TEM and they reported that the ferrite grain size in the annealed microstructure was not uniform, and a finer ferrite grain size was found in the more highly deformed zone. This is also an indication that deformation can provide more nucleation sites and results in a more refined structure.

Besides in the nucleation stage, the cementite particles can also be expected to play an important role in retarding the growth of recrystallized ferrite grains. As seen in Fig. 85, the spheroidization process of carbide lamellae took place well before recrystallization in the course of the annealing of deformed pearlite, so that cementite particles are readily available for the retardation.

![Fig. 85. The spheroidization process during the annealing of deformed pearlite at 650°C for (a) 20 min (b) 30 min (c) 50 min (d) 90 min.](image)

Fig. 86 shows the distribution of cementite particles in partially and fully recrystallized structures. Cementite particles seem to be located on the grain boundaries, where they can
effectively retard the grain boundary movement. It is also obvious that the size of cementite particles increases with a prolonging of the soaking time.

Fig. 86. The dispersion of cementite particles during the annealing of deformed pearlite (a) in partially recrystallized microstructure (b) in fully recrystallized microstructure. 600°C, 1 hour.

Second phase particles can have a great influence on the recrystallized grain size. The Zener drag and solute effects are well-known for a long time and reported in the literature. However, recently it has been observed that experimental results would indicate a significantly higher drag force than predicted from the classical Zener model [98]. This is because the number of particles in contact with a moving boundary at any time is higher by a factor of 3 to 4 than given by the original Zener rigid boundary estimate. In any case, the main parameter controlling the grain size in annealing is the ratio of Fv/r (the volume fraction Fv of small particles and their mean radius r) [129]. This will be discussed more in Section 4.3.2. Fv/r reflects the Zener drag and affects the number of viable recrystallization nuclei and also the grain size (D_{LIM}), at which the normal grain growth
will stagnate. Fig. 87 shows schematically the effect of particle dispersion on recrystallized grain size [129].

![Fig. 87. The effect of particle dispersion on recrystallized grain size[129].](image)

The curve $D_N$ is the grain size after the primary recrystallization affected by PSN. The number of nuclei from large particles or any other sites will decrease as $Fv/r$ increases. At a certain dispersion level ($Fv/r=B$), the number of nuclei becomes effectively zero, and then any recrystallization cannot occur. The curve $D_{LIM}$ is the grain size at which the normal grain growth will cease. The point at which these two grain sizes are equal is denoted by $Fv/r=A$. In the regime of $Fv/r < A$, the grain size after the primary recrystallization is determined by the number of available nucleation sites, and if this is large, it may be very small, but grain growth is possible. Also, in the regime of $A < Fv/r < B$, the material recrystallizes to a grain size $D_N$, determined by the number of nuclei. However, this is above the value of $D_{LIM}$ and therefore normal grain growth will not occur. In the regime of $Fv/r > B$, the Zener pinning is sufficient to suppress discontinuous recrystallization and the particles stabilize the deformed or recovered microstructure.

The situation in the deformed pearlitic microstructure with lamellae is different from the case shown in Fig. 87. In this figure it is assumed that all nucleation sites come from PSN, and the number of nucleation sites will decrease with increasing particle diameter. However, in the deformed pearlitic microstructure with the cementite lamellae, deformation has two important effects, to increase the recrystallization nucleation sites by the PSN mechanism and simultaneously to increase the pinning force. As mentioned above, as long as the particle size is bigger than 200 x 10 nm they play a role by PSN [128]. During the deforming of pearlite, the cementite lamellae were broken into plate-form pieces, their size decreasing with increasing strain. Since $Fv$ is a constant for a given pearlitic structure, the value of $Fv/r$ increased with the increasing strain and the Zener drag increased, too.
There is hardly any doubt that the cementite particles in deformed pearlite of 0.8C steel, formed by spheroidization during annealing, can retard the grain growth. The recrystallized microstructure was very stable, so that, for example, a ferrite grain size of 0.75 µm, shown in Fig. 49, remained constant at least for 1 hour at 600°C. This will get further support from the thermal stability results of high-carbon steels of 0.3C, 0.4C and 0.6C, to be described in Section 4.3. This means that, according to Fig. 88, Fv/r must be beyond A. Whether Fv/r > B, it might be a question. The recrystallized microstructure of deformed and annealed pearlite (see Figs. 86 and 87) resembles exactly that formed by the extended recovery, where the second phase particles are located on the grain boundaries, as schematically shown in Fig. 88 [130]. In the extended recovery, the migration of low and high-angle boundaries is impossible and normal discontinuous recrystallization is inhibited by the particles, so that the subgrains can only grow with the coarsening of particles in the course of annealing.

Furthermore, it has been shown that a stable subgrain structure can form in the following conditions: interparticle spacing is less than ~1 µm or the ratio Fv/r is greater than ~ 0.2 µm⁻¹ [130]. From Figs. 85 and 86, the carbide spacing seems to be on an order of 1 µm (note that the deep etching used to reveal the ferrite grain size means a non-planar surface so that the view overestimates the number of particles on a plane section) and the ratio Fv/r is also greater than 0.2 µm⁻¹ (an estimate: Fv/r = 0.09 / 0.25 µm ≈ 0.36 µm⁻¹). This value is in reasonable agreement with the data of Ohmori et al. [83] proposing Fv/r ≈ 0.5 µm⁻¹ for a 0.3%C steel (UFF + cementite). As a summary, it can be concluded that the cementite particles play an important role on both particle stimulated nucleation and on the retarded growth of recrystallized ferrite grains in the deformed pearlitic microstructure providing a very efficient grain size refinement.

![Schematic microstructure formed by extended recovery](image)

Fig. 88. Schematic microstructure formed by extended recovery (a), where the grain growth is controlled by the coarsening of second-phase particles (b) [130].

### 4.2.2 Refinement mechanism in annealing of deformed martensite and tempered martensite

As is well known, lath martensite has a three-level hierarchy in its morphology: (I) Lath; a single crystal of martensite containing a high density of lattice defects, (II) Block;
aggregates of laths with the same crystallographic orientation, and (III) Packet; aggregates of the blocks having the same habit plane [131]. Within one prior austenite grain, there can be several packets due to four different \{111\}_γ planes in the austenite. Each packet includes several blocks composed of laths with different variants lying parallel to the same \{111\}_γ plane. According to K-S relationship, it is possible to have 24 different varieties in an identical prior austenite grain. It is estimated that 83% of the block boundaries and the packet boundaries can be of a high-angle character [131]. It means that martensite is a kind of fine grained structure subdivided by a number of high-angle boundaries. In addition, martensite includes a high density of dislocations as well as a number of solute carbon atoms in the as-transformed state.

The deformation modes of martensitic structure in iron alloys is often thought to be analogous to that of b.c.c iron [132, 133]. The tetragonal martensite clearly introduces the possibility of new deformation modes, in addition to those operative in pure b.c.c structures. Cubic martensite deforms by slip at room temperature, and it remains on the main mode of plastic deformation down to 77 K. In the range of 0.05-0.4%C, deformation is increasingly produced by mechanical twinning rather than slip, and for carbon contents ≥0.4% only twinning is observed. Slip lines in low alloy martensites are often wavy and irregular at room temperature, and single crystal studies on b.c.c metals have shown that this is due to the pronounced tendency for screw dislocations to cross-slip [134].

Ueji et al. [131] classified the details of the morphology at the interior of deformed (at 50% reduction) martensite in a low carbon (0.13%C) steel into three kinds of microstructure:

* A: very fine lamellar structure mainly elongated parallel to rolling direction,
* B: irregular bent lamellar structure,
* C: lump of martensite laths with shear bands.

In fine lamellar structure, there are many ultrafine lamellar dislocation cells. Similar lamellar structures have been previously observed in materials intensively strained by conventional rolling or accumulative roll-bonding [58]. In the irregular bent lamellar structure, a bunch of laths is bent to orient in various directions, and numerous dislocations exist here. There is a very high density of dislocations in the lump of martensite laths with shear bands. It is found that the submicronstructure, the mean distance between the boundaries, for instance, in the very fine lamellar structure after the 50% deformation is similar to that of ferrite formed by strains larger than 4.

It is known that in metastable austenitic stainless steels, ultrafine austenite grains well below 1 μm are obtained in the reversion process from heavily deformed (order of 50%) strain-induced martensite [80]. A high deformation of lath martensite is thought to be of vital importance for the formation of submicron scale grains in these steels. Only when the lath-martensitic structure is completely destroyed by heavy pre-cold-working, equiaxed austenite grains can nucleate at random and grow into the recovered martensitic matrix. In agreement, Tokizane et al. [135] showed that fine grained austenite is formed in heating from heavily (75-84%) deformed lath martensite in 0.2%C steels, while coarse austenite grains form from non-deformed martensite. Ueji et al. [131] obtained a UFF structure (180 nm) with nanocarbides by an annealing of the cold-rolled martensite in a 0.13%C steel even with the reduction as low as 25%, although the fraction of UFF among carbides and tempered martensite increased with increasing reduction. The reason for the effective
refinement was, according to them, mainly related to the initial fine-grained martensitic microstructure, the mean grain size of blocks/packets being estimated as 3.2 μm.

It is generally accepted that the recrystallization is difficult to occur in non-deformed martensite in spite of its high dislocation density (about $10^{10}$-$10^{11}$/cm$^2$) compared to that in deformed ferrite [136, 137, 138, 139]. This has been attributed to the existence of fine carbides precipitates, which suppress the grain boundary migration and therefore retard the recrystallization [140, 141]. However, in addition to such a retarding effect of carbides, the difference in the arrangement of dislocations between martensite and deformed ferrite must be essentially related to the difficulty of recrystallization of lath martensite. The dislocations in martensite are distributed uniformly, while plastic deformation such as cold rolling is usually inhomogeneous, resulting in the formation of deformation bands and a dislocation cell structure in the ferrite. The driving force for recrystallization in a lath martensite structure maybe somewhat lower than that in deformed ferrite. Moreover, the inhomogeneity of deformation by cold rolling provides effective nucleation sites for recrystallization.

In the present work, opportunities to obtain UFF by annealing of deformed martensite (the SRM route) in C-Mn, Nb, Nb-Ti and Nb-hTi steels were investigated (Section 3.3). Here, the primary importance of deformation was also confirmed. In experiments performed it was clearly observed that in C-Mn, Nb, Nb-Ti and Nb-hTi steels with the martensitic microstructure, if the martensite was directly annealed without any deformation, UFF was not obtained but coarse grained ferrite, instead. Secondly, different refinement efficiencies were found in these steels, as shown in Figs. 52~55. At a 70% reduction, ferrite grain sizes of about 1 μm were obtained only in C-Mn and Nb steels, while even at a reduction of 90%, ferrite grain sizes in Nb-Ti and Nb-hTi steels were only refined to 2.5 μm. Hence, besides a deformed martensitic microstructure, the chemical composition also affects the formation of UFF. It seems that the influence of the particles precipitated during the annealing cannot be neglected.

Precipitation-recrystallization interactions are well known to happen in TMCP, where strain-induced precipitation of NbCN even prevents recrystallization in the austenite, so that work-hardened pancaked grains can be obtained in the finishing rolling stage. The particle dispersions can affect even the recovery stage [142]. Particles exert a retarding pressure (the Zener-drag) on the moving subgrain boundaries, so that the subgrains will be prevented from reaching a critical size necessary to become potential nuclei for the recrystallization. As a consequence, the structure prior to cold rolling or formed during deformation is maintained during annealing.

The progress of the precipitation process in the C-Mn, Nb, Nb-Ti, and Nb-hTi steels during annealing can be seen in Figs. 89-92. The precipitate particles always became visible already during the recovery stage in all the steels, and the number of particles was higher in the C-Mn and Nb steels than in the Nb-Ti and Nb-hTi steels.
Fig. 89. Microstructure development in C-Mn steel during annealing of deformed martensite at 600°C (a) 10 min (b) 30 min (c) 40 min (d) 50 min.

Fig. 90. Microstructure development in Nb steel during annealing of deformed martensite at 600°C (a) 10 min (b) 30 min.
Fig. 91. Microstructure development in Nb-Ti steel during annealing of deformed martensite at 600°C (a) 10 min (b) 20 min (c) 30 min (d) 60 min.

Fig. 92. Microstructure development in Nb-Ti steel during annealing of deformed martensite at 700°C (a) 10 min (b) 40 min.

The coarsening of cementite particles in C-Mn steel can be seen in annealing at 550°C in Fig. 93, and it can be estimated that the particle diameter became almost doubled from 0.12 µm (in 10 min) to 0.22 µm (in 100 min). Contrary to this, the precipitate diameter in Nb-Ti steel remained almost constant even in annealing at 600°C, as shown in Fig. 94. Ohmori et al. [83] measured the carbide diameters in UFF low-carbon steels and the values were from 0.22 to 0.16 µm for steels with 0.1 to 0.3%C, respectively, i.e. somewhat
coarser than the present particles. In annealing above 600°C, some coarsening took place, so that the diameter in the 0.1%C steel was 0.3 µm after 1 h at 700°C.

Fig. 93. The variation of cementite particles in C-Mn steel in annealing at 550°C (a) 10 min (b) 30 min (c) 50 min (d) 100 min.

Fig. 94. The carbide particles in Nb-Ti steel in annealing at 600°C (a) partially recrystallized microstructure, 40 min (b) fully recrystallized microstructure, 100 min.

The differences in the coarsening kinetics in different steels can be understood on the basis of the diffusion rates of different precipitation-rate controlling elements. In ferrite, the diffusion rates of Ti and Nb atoms are much lower than that of carbon. For instance, at 700°C, the diffusion coefficient of carbon is $3.0 \times 10^{-11}$ m$^2$/s, while that of Ti is only...
3.66 \times 10^{-17} \text{ m}^2/\text{s}, and that of Nb is even lower [143]. Therefore, the precipitation rate of cementite particles in C-Mn steel was much faster than that of microalloy carbide particles in Nb-Ti and Nb-hTi steels. Because the volume fraction of precipitated particles is controlled by the diffusion rate and supersaturation, a higher fraction can precipitate in a given time in C-Mn steel than in Nb-Ti or Nb-hTi steels. However, Nb-steel also showed a fast coarsening kinetics of precipitates. This can be explained on the basis of the high carbon content of this steel (0.15%C). The ratio of Nb:C 0.033/0.15 = 0.22 is low so that a major part of carbon remains unbound by Nb and it can form cementite in addition to NbC. Hence, its behavior resembles that of C-Mn steel.

As already mentioned above and in the discussion in the previous section, particles can play an important role on the subgrain and recrystallized grain sizes in particle-containing alloys. A fine particle dispersion may exert a strong pinning effect on subgrains, and a stable subgrain structure can form in some cases. The recrystallization process in a particle-containing alloy, as schematically shown in Fig. 95 (the vertical axis represents the orientation, the horizontal direction the distribution of second particles and boundaries), can be classified into continuous recrystallization and discontinuous (normal) recrystallization [142]. If the spacing of high-angle boundaries approaches the interparticle spacing (A/B~1), the annealing process will occur by the mode of discontinuous recrystallization, and a structure, which contains mainly high-angle boundaries, may be formed without any particle coarsening, as schematically shown in Fig. 95(f). If the spacing of high-angle boundaries is much less than the interparticle spacing (A/B>1), the annealing process will be in the mode of continuous recrystallization, in which the recovery occurs, and the initial subgrain growth will be largely unaffected by the particles until the subgrains have grown to a size comparable with the interparticle spacing, as the case shown in Fig. 95(b). At this stage, the recovery structure is stabilized. The orientation spread within a grain is not likely to change significantly during the subgrain growth, and as there is no overall orientation gradient within the grains, the subgrain misorientations remain small. Further annealing at the higher temperature may coarsen the particles that may allow the subgrains to grow, as shown in Fig. 95(d), and finally the microstructure will comprise mainly high-angle boundaries.
The different refinement efficiencies observed in the four steels tested here can be explained as the different retardation effects during the annealing of deformed martensite. According to the Zener model, the limiting grain size, $D_z$, is related to the ratio of $F_v/r$ ($F_v$ the particle volume fraction and $r$ is the radius of the spherical particle), as expressed by a typical formula [130]:

$$D_z = \frac{4r}{3F_v}$$  \hspace{1cm} (11)

As concluded earlier, in the C-Mn and Nb steels, the precipitates were cementite and in the Nb-Ti and Nb-hTi steels they were NbTiC (presumably also nitrides). The estimated values for $F_v/r$ from Figs. 89–94 are summarized in Table 8. For the C-Mn and Nb steels they are simply based an assumption that the carbon content determines the cementite fraction. For the Nb-Ti and Nb-hTi steels, the Nb and Ti contents also have to be accounted for. In high-Ti steel only part of Ti is free for fine carbonitride precipitation. From the experimental data of Ohimori et al. [83] for low-carbon steels, it can be determined that the cementite volume fraction (in %) is 12 times the carbon content of the
It is reported that the typical driving force for grain coarsening is about 0.1 MPa, while the typical driving force for recrystallization is approximately 20 MPa, i.e. about 200 times larger than that for grain growth [144]. Driver [98] gave the value of 0.5 MPa for micrometer-sized material. It is known from TMCP that only the strain-induced precipitation, NbCN at a size of 10 nm or so, can prevent recrystallization, but the grain growth can be prevented by much coarser particles [144]. Hence, it can be concluded that recrystallization cannot be prevented by the particles 100-150 nm in diameter in the four steels tested, as also evident from Figs. 52~55, and Figs. 89~92.

In order to further confirm the effect of cementite particles on the recrystallized grain sizes in the SRM route, three high-carbon steels of 0.3C, 0.4C and 0.6C were also tested. Since high-carbon martensite is impossible to be deformed, the martensitic microstructures were tempered before the deformation and subsequently annealed. In annealing, a high refinement efficiency was observed in all these steels, as shown earlier in Fig. 57~59 (Section 3.3). Because they did not contain any microalloying elements, the higher refinement efficiency should be a result of the influence of cementite particles. Figs. 96~99 show that a large number of cementite particles are formed in these steels, and their number is basically proportional to the carbon concentration. The diameter of cementite particles in the fully recrystallized microstructure seem to be almost constant, mainly within the range of 100 ~ 200 nm, as seen in Figs. 57~59 and Figs. 96~99. Therefore, the ratio of Fv/r for these high-carbon steels is higher than that for the C-Mn steel.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Estimated volume fraction, %</th>
<th>Measured mean diameter, µm</th>
<th>Fv/r, µm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Mn</td>
<td>1.1</td>
<td>0.15</td>
<td>0.147</td>
</tr>
<tr>
<td>Nb</td>
<td>1.8</td>
<td>0.15</td>
<td>0.24</td>
</tr>
<tr>
<td>Nb-Ti</td>
<td>0.2</td>
<td>0.12</td>
<td>0.03</td>
</tr>
<tr>
<td>Nb-hTi</td>
<td>0.05</td>
<td>0.10</td>
<td>0.01</td>
</tr>
</tbody>
</table>
Fig. 96. The progress of precipitation of cementite and recrystallization of ferrite during the annealing of deformed tempered martensite at 600°C in 0.3C steel. (a) 40 min (b) 100 min.

Fig. 97. The precipitation of cementite and recrystallization of ferrite during the annealing of deformed tempered martensite at 600°C in 0.4C steel. (a) 5 min (b) 150 min.

Fig. 98. The precipitation process of cementite and recrystallization of ferrite during the annealing of deformed tempered martensite at 600°C in 0.6 steel. (a) 40 min (b) 100 min.
Fig. 99. The growth of cementite particles in the annealing of deformed tempered martensite at 600°C in 0.4C steel (a) in the initial microstructure (b) 10 min (c) 20 min (d) 60 min.

However, in high-carbon steel of 0.6C, the initial microstructure of martensite is not lath martensite, but twinned martensite, and its plastic deformation behavior is different from that in low-carbon steels, as mentioned before. Furthermore, due to tempering, carbides are formed before the deformation, so they may results in more intensive non-homogeneous local plastic straining. There may be other reasons for a higher refinement efficiency, but anyhow, they do not dismiss the conclusion that a bigger amount of second phase particles can result in a higher refinement efficiency in the SRM route.

It can be noted here that UFF structure could not be obtained in C-Mn, Nb, Nb-Ti, or Nb-HfTi steel from deformed tempered martensite when a low tempering temperature of 300°C (1 hour) was used. The reason was not carefully investigated, but it could be related to a low-temperature tempering. It resulted in a large number of fine precipitated particles in the deformed martensite microstructure that can exert a pinning force so high that the recrystallization is retarded. Recrystallization cannot take place before a sufficient coarsening of the precipitated particles. Tempering before a deformation will also result in a reduction of the dislocation density, so that at a given deformation, the number of nucleation sites will decrease.
4.2.3 Refining mechanism in annealing of deformed bainite

In steel, bainite is a product of a non-lamellar, non-cooperative mode of eutectoid decomposition of ferrite and cementite [145]. The nature of bainite, from lath size and shape to carbide size and distribution, varies systematically with the temperature of transformation. The dislocation density of the laths increases with a decreasing transformation temperature. The morphology of low bainite is similar to that of lath martensite, composing of long ferrite lathes. A striking microscopic characteristic of lower bainite is the growth of carbide rods within the ferrite plates. The carbide can be either cementite or ε-iron carbide, depending both on the transformation temperature and on the composition of the steel. In upper bainite, ferrite has a much lower carbon concentration (<0.03%C), and carbide precipitation does not occur within the laths. The morphology of cementite formed on the lath boundaries is dependent on the carbon content of the steel. In low-carbon steels, the carbide will be present as discontinuous stringers and isolated particles along the lath boundaries, while at higher carbon levels the stringers may become continuous. In some steels, the enriched austenite does not precipitate carbides but remains as a film of retained austenite. This type of bainite is often referred to as granular bainite. The lower bainite appears more acicular than the upper bainite, and the ferrite subunits are about 0.5 µm wide and slightly disoriented from each other. The plates possess a higher dislocation density than in upper bainite, but not so much as the martensite of similar composition.

Based on the results and analysis from deformed martensite and pearlite, the deformed bainite microstructure can also be expected to have a high potential refinement efficiency. First, there exists fine cementite particles, and secondly a similar internal microstructure to martensite, cells with subgrain boundaries.

Here, the bainite microstructure formed at 350°C in the high-carbon steel of 0.6C, was deformed to the strain of 1.2, and after annealing, the microstructure with an UFF grain size of about 0.8 µm was successfully obtained, as shown in Fig. 51. The high-carbon bainite formed at the isothermal holding at 350°C belongs to the type of upper bainite and so that its microstructure can be expected to offer similar nucleation sites as lath-martensite does. The upper bainite has cementite particles along the lath boundaries that can play an important role on pinning the grain boundaries during the annealing of deformed bainite.

Fig.100 shows the coarsening of cementite particles during the annealing of deformed bainitic microstructure of 0.6C steel. Comparing with the annealing process of deformed tempered-martensite of the same steel, it can be seen that similar structures regarding the final recrystallized grain size and the cementite morphology, including the distribution and the particle diameter, were obtained.
Because of the big cementite particles in bainite, it is reasonable to think that not only the recrystallized grain growth but also the subgrain growth will be affected by these particles during the annealing of deformed bainite analogically as in martensite, as discussed earlier.

In order to further ascertain the vital role of carbide particles on recrystallized grain growth, a ULC steel (Table 1), was selected to investigate whether a UFF structure can also be obtained from deformed carbide-free bainite. The specimen was deformed at a the strain of 1.2 and subsequently annealed at 600°C for various times. A typical microstructure in the early recrystallization stage can be seen in Fig. 101, where it is seen that the recrystallized grains tend clearly to inherit the size of the prior austenite grain size and they are larger than 5 µm. The final ferrite grain size in the fully recrystallized microstructure was even larger than 40 µm.
Fig. 101. Ultralow carbon 0.006C steel microstructure with first recrystallized grains when deformed at a strain of 1.2 and subsequently annealed at 600°C for 60 min.

Since the carbon content in ultralow-carbon steel was so low that practically no precipitation could occur during annealing, no pinning effect was exerted on the recrystallized grain growth. Also, the number of nucleation sites is low, probably only on the prior austenite grain boundaries. These factors explain the coarse grain size formed. Hence, it seems that the retardation effect on the primary recrystallized grains during the static recrystallization of ferrite is a very important factor in obtaining a high refinement efficiency.

4.3 Thermal stability in annealing and welding

4.3.1 Annealing of the UFF structure

As mentioned in the introduction, owing to a high stored energy (a driving pressure of about 0.5 MPa) [146] in the grain boundaries, UFF grained materials are often reported to be more prone to grain coarsening and the grain growth is observed even at relatively low temperatures [146, 147], or below the conventional recrystallization temperatures of coarser grained materials [146, 148, 149]. For instance, Shin et al. [100] found coarsening of 0.2 µm grain-sized severely deformed ferrite in an 0.15C-1.1Mn steel, obtained by the ECAP method (strain ~ 4), above 783 K (510°C) in connection of recrystallization and an enhanced spheroidization of the cementite phase. The V-bearing steel was found to be somewhat more stable[150]. Priestner and Ibraheem [49, 151] reported some abnormal grain growth at 600°C after a 20 min holding in a Nb-bearing UFF steel strip, processed by 66% cold-rolling reduction. Ohimori et al. [152] found abnormal grain growth to start in 1 hour at 600°C in a multi-pass warm caliber-rolled 0.15%C steel with a grain size of
0.4 µm. Ueji et al. [62] observed a drop in strength and recovery of the ductility in annealing above 550°C for 30 min in UFF microstructure, processed by the SRM route. The yield strength and evolution of microstructure are shown in Fig. 102. However, in their study, coarse grains appearing at 600°C meant a grain size of a few micrometers, only. As a maximum stability, Hodgson et al. [26] noted that micrometer-size grains were stable even after 30 min at 700°C. Xu et al. [153] observed that ultrafine grains can be stabilized by a fine precipitation in an Al-Zn-Mg-Zr alloy up to temperatures of 0.83Tm (Tm=melting temperature in K), demonstrating a very high Zener drag achievable. Driver [98] has most recently discussed the matter.

Fig. 102. Changes in the yield strength (a) and UFF grain structure (b) obtained by the SRM route in Fe-0.13% C steel in annealing at various temperatures [62].

The present results indicated that UFF grained microstructures with a grain size in the range of 1-3 µm were quite resistant to grain coarsening, both in the C-Mn and microalloyed steel grades. For instance, the UFF grained microstructures of the C-Mn, Nb-Ti and Nb-hTi steels, obtained by the DIF route, remained stable even up to more than 2 hours at 700°C. The three high-carbon steels of 0.3C, 0.4C and 0.6C also presented a good thermal stability, for the ferrite grains only showed insignificant coarsening up to 4 hours at 650°C. However, the UFF grained microstructures of the C-Mn, Nb-Ti and Nb-hTi steels obtained by the SRM route showed a weaker thermal stability (Figs. 61, 63 and 65), for the normal grain growth occurred during isothermal soaking at 600°C in the grain structure of a few micrometers in size.

The thermal stability of UFF grained steels has been reported to be influenced by the processing route [154]. Here, it seems that there are two different modes of grain coarsening in UFF grained microstructures, normal and abnormal grain growth. These grain growth modes do not relate with the chemical composition of the steel, for even in the same steel the grain growth can occur in both modes. For instance, for the C-Mn, Nb-Ti and Nb-hTi steel, the grains in the DIF specimens grew in the abnormal mode, while in the SRM specimens in the uniform mode. The grain growth mode in UFF microstructures is neither related to the method of processing, for in the SRM specimens of C-Mn, Nb-Ti and Nb-hTi steels, the normal grain growth occurred, while in three high-carbon steels of 0.3C, 0.4C and 0.6C, the growth was an abnormal one.
The distribution of carbide particle in the recrystallized microstructure of SRM specimens is different between the high-carbon steels of 0.3C, 0.4C and 0.6C and the four low-carbon steels of C-Mn, Nb, Nb-Ti and Nb-hTi, as evident from Figs. 52~55 and Figs. 57~59. In the low-carbon steels, the carbide particles precipitated from the lath-martensite were distributed uniformly, not only on the grain boundaries, but also in grain interiors. Contrary to this, in the high-carbon steels the carbide particles precipitated from the twinned-martensite were located on the grain boundaries or corners. In all the steels, the particles were found to precipitate before recrystallization, and therefore the particles can have an influence on the subgrain formation. It has been reported that a limiting stable subgrain can form in the following conditions: the interparticle spacing is less than ~1 µm, or the ratio volume fraction/particle size $F_v/r$ is greater than ~ 0.2 $\mu$m$^{-1}$ [130]. In the recrystallized microstructure of steels 0.3C, 0.4C and 0.6C, the interparticle spacing seems to be on an order of 1 µm, but $F_v/r$ is greater than 0.2 $\mu$m$^{-1}$ ($r$ is less than 0.1 µm even in the recrystallized microstructure, and $F_v$ was estimated to be not less than 2%). Therefore, the particles can restrict the subgrain growth by pinning the boundaries and prevent the grain growth. As with the case shown in Fig. 88, in the course of annealing of deformed pearlite, the recrystallization was controlled by the coarsening of particles. Contrary to this, in the low-carbon steels of C-Mn, Nb, Nb-Ti and Nb-hTi, the precipitate interspacing is longer than 1 µm and also the $F_v/r$ is smaller than 0.2 $\mu$m$^{-1}$ (Table 8). Therefore, the subgrains cannot be pinned so firmly as in the case of the three high-carbon steels, and subgrain boundaries can escape from the precipitate pinning and surpass some particles, and as a result, some precipitate particles are located inside the grains as well, as evident from Figs. 90~95. Some normal grain growth is then possible.

In the course of annealing, the four steels, C-Mn, Nb, Nb-Ti and Nb-hTi, have somewhat different particle coarsening behaviors due to different chemical compositions. The precipitates in C-Mn (0.097%C) and Nb (0.15%C, 0.033%Nb) steels are cementite, but in Nb-Ti (0.072%C, 0.046%Nb, 0.011%Ti) and Nb-hTi (0.074%C, 0.048%Nb, 0.13%Ti), the precipitates are TiNb(C,N). The high carbon content of 0.15% in Nb steel is the reason for the presence of cementite in this steel, for the 0.033%Nb cannot bind all of the carbon. As is well known, microalloy carbides have a higher thermal stability compared to cementite, so that the particle coarsening rate is lower and they can keep a higher retardation on the grain boundaries compared to that in C-Mn and Nb steels.

However, in the DIF specimens of four C-Mn, Nb, Nb-Ti and Nb-hTi steels with a grain size finer than 2 µm, the grain growth took place in an abnormal mode. The reason could be related to the influence of precipitate particles and in this case, to the nonuniform distribution of them. After DIF processing, a dual-phase structure with UFF and carbide aggregate/martensite was present. In annealing, their structure will consist of UFF ferrite and cementite particles in the tempered matrix in separate regions. Therefore, grain growth can occur by the secondary recrystallization mechanism starting in areas without particles and low pinning.

The present results demonstrate that ultrafine microstructure in steels can be thermally stable up to temperatures of about 0.6Tm, obviously due to the dispersion of carbide particles present, but in very reasonable numbers and sizes, presumably without detrimentally affecting mechanical properties.
Mechanical properties of welded joints are of importance when assessing the applicability of UFF steels for steel structures. Because of this special fine-grained microstructure, it can be assumed to have a high tendency to coarsen and deteriorate the properties. The weldability of UFF grained steels has been investigated to a limited extent previously, but softening has been observed to occur at the HAZ even in laser welding [105, 106, 155]. Hence, the control of the width of the softened zone in the HAZ may be practical way for industrial applications of UFF grained steels.

As described earlier in Section 3.4, the present annealing results indicated a high thermal stability of the UFF structure in C-Mn, Nb-Ti and Nb-ATi steels. The SRM route might be a practical route to manufacture UFF grained steels, and thin rolled and annealed strips were available for welding tests, so that it was decided to investigate briefly the weldability of UFF steels obtained by this route. The test materials were small pieces of thin sheets, on an order of 1 mm in thickness, and therefore only special low-heat input welding methods were employed, electron and laser beam welding.

As described earlier, the hardness measurements across the welded seams and microstructure examination did not reveal any softening. The hardness both in the welded metal and the HAZ even became higher than in the base metal. This is evidently a consequence from a high cooling rate in the weldments and the formation of bainite or martensite from the decomposed austenite. Therefore, the hardness is not directly related to the ferrite grain size. However, from a very fine microstructure that was extremely difficult to analyze in an OM, it can be concluded that at low heat inputs, the grain coarsening at HAZ, if any, remains insignificant in these UFF grained steels. Of course, the weld metal columnar grain structure is quite coarse and it does not inherit anything from the original fine-grained structure.

As comparing with some published welding results, where softening occurred in the HAZ of a thick plate, it must be noted that in those experiments the heat input used has been several times higher, as in Fig. 103, for instance. There the hardness has dropped below 200 HV in the softest zone corresponding to a grain size larger than 2 µm. If we assume, as discussed earlier, that grain coarsening is inhibited by the carbide particles, the particle stability is of concern. As is well known, cementite and even microalloy carbides will dissolve in heating of the austenite so that they will be absolutely lost at the HAZ adjacent to the fusion line. The width of the softened zone is then an important factor, similarly as in the welding of ordinary TMCP steels. Also, the selection of a filler metal has to be considered for the welding of UFF grained plates and sheets.
Fig. 103. The influence of heat input on the hardness of the HAZ in a 0.05% C-2.03% Mn steel plate with an original ferrite grain size smaller than 1 µm [155].
5 Recommendations for future work

This work was only the first stage for investigating the potential of the processing of UFF steels. The two routes were shown to be successful to produce UFF microstructures and the influencing factors were revealed. However, no assessment of the relevance of these factors as considering the practical steel manufacturing processing has been performed. In order to continue in the development work of UFF steels, such a discussion should be performed with industry experts to define the future tasks to be undertaken in order to clarify the real routes convenient for bulk production in a steel mill.

In microstructural studies, the EBSD technique could and should be utilised much more than in the present study.

In this work the mechanical properties of the UFF structures were not tested at all. This should be performed and then special efforts could be directed to dual-phase structures, such as UFF-martensite, which seems to show enough ductility. Also, the possibility of utilising the bimodal grain distribution could be investigated.

The weldability of the UFF structure was tested at very low heat inputs and thin sheet thickness only. More extensive investigation would be required to assess the properties achievable, especially in arc welded joints.

A detailed analysis of the most recent state of the art of UFF metals in a huge number of papers published should be carried out.
6 Summary

It has been shown in this work that the grain size of ferrite can be refined to 1-3 µm, called the ultrafine ferrite (UFF) grain size, by two methods: the dynamic transformation of ferrite from a heavily hot-rolled austenite (deformation-induced dynamic transformation, the DIF route) and the static recrystallization annealing of severely cold-rolled ferrite or other initial microstructures (the SRF/SRM route). The principles of both routes have been previously reported in the literature but they were studied more extensively here, and particularly the refinement mechanisms were discussed in detail.

The DIF route UFF grained ferrite could be obtained in all of the four low-carbon steels tested: C-Mn, Nb, Nb-Ti and Nb-hTi steels. In this route, the final grain size of ferrite is influenced by many processing variables, as also reported earlier in the literature. This investigation confirmed that:

1. Higher strain applied results in a finer ferrite grain size and in higher deformation-induced ferrite fractions. Minimum strains required for UFF are on an order of 0.9 - 1.2.
2. Deformation at a low temperature, just at the A_{\text{f3}}, results in finest grain sizes.
3. Strain rate has only a minor effect on the ferrite grain size, at least if between 0.1 and 10 s\(^{-1}\).
4. Strain rate has a more important influence on the type of the second phase present in addition to the strain-induced ferrite, carbide aggregate or martensite, especially in the case of lower deformation-induced ferrite fractions.
5. Refining the prior austenite grain size highly enhances the ferrite grain size refinement, and it is quite crucial in practice. It can be obtained by cold rolling the sheet before the low-temperature reheating and by repeating the reheating cycle or by combining them both. Also, an initially martensitic structure for cold-rolling can be utilised.
6. Somewhat finer grain sizes were obtained in higher microalloyed steels than in the C-Mn steel.
In the cold rolling-static recrystallization route (SRF/SRM), various starting microstructures were investigated to produce the UFF grain size, and the main results can be summarised as:

(7) In the steel with a single-phase ferrite microstructure, UFF cannot be obtained in this route.

(8) From deformed pearlite, UFF can be obtained in annealing.

(9) From deformed bainite, UFF can be obtained in annealing in a high-carbon steel, but not in a ultralow carbon steel. Hence, some carbide particles seem to be necessary to stabilise the grain size.

(10) From deformed martensite, UFF can be obtained in annealing. The refinement effectiveness depends on the presence of precipitated second phase particles before the recrystallization process. The volume fraction and the diameter of particles can affect the recrystallized grain size, so that the finer and higher the volume faction of particles, the finer is the ferrite grain size. Grain sizes well below 1 µm were obtained in high-carbon steels.

(11) From deformed tempered martensite, UFF can be obtained in steels with 0.3-0.6%C, for in these steels, the carbides can suppress the grain growth. However, UFF grain size was not obtained from the low-carbon martensite tempered at a low-temperature (300°C).

Concerning the thermal stability of the UFF structure it was observed that:

(12) UFF microstructures obtained by the DIF route have a very good thermal stability in all the steels investigated. A high Zener drag caused by cementite particles is an obvious reason for this. The grain growth begins at 700°C after a holding as long as 150-240 min in low-carbon steels, and then it occurs in the abnormal mode. This is presumably due to a nonuniform distribution of carbides in the annealed dual-phase structure of strain-induced ferrite together with the carbide aggregate or martensite.

(13) UFF microstructures obtained from deformed martensite have a thermal stability dependent on the chemical composition. In low-carbon steels, the UFF microstructure coarsened in annealing by the normal grain growth mode at 600°C, while in the high-carbon steels, the UFF grain size remained unchanged for several hours at 600°C before the abnormal growth started. It was shown that the UFF stability is determined by the volume fraction and the size of the precipitated particles. In the tempered steels, the carbide particles are uniformly distributed and the grain growth can proceed with the coarsening of particles.

(14) In welding at low-heat inputs, such as in electron beam and laser welding methods, no coarse-grained zone was formed and no softening took place at the heat-affected zone in the low-carbon steels tested. Hardness increased from the base metal towards the heat-affected zone and the weld metal owing to harder phases formed in fast cooling. According to the literature, at higher heat inputs some softening may be unavoidable.
References


