

Thermomechanische Behandlung

Experimente





Experiments and Results





Inserting Sample into pneumatically Transport System









Transport to Dies





Course of a Hot Working Simulation





WUMSI-Forging Simulation























Û





Secondary Specimen





Secondary Specimen









Cooling Rates









(small samples: $\dot{T} < 400 \text{ K/s}$)



Heavy Warm Rolling



Cost saving alternative of quench and tempering





Ferrite grain boundaries enhance the spheroidization







Increased cooling rate (finer lamellae) enhance the spheroidization



Heavy Warm Rolling



Spheroidization of pearlite in steel with 0.66% C (deformation at 610°C)



Introduction: Properties





Introduction: Properties





after Nagai



We have some problems ...



High demands on the novel UF routes



Niikura et al.

- I. Deformation of undercooled γ
- II. Deformation in the state of multiple phases
- III. Inverse transformation after heavy deformation



very difficult for large scale industrial production





- obtain ultra fine grain in plain C-Mn steel (lower cost elements, easy recycling)
- fine cementite dispersion
 (increase strain hardening, stabilization)
- relationship between microstructure and mechanical properties of UF grained steel
- industrial applicability (industrially applicable process parameters)



	С	Si	Mn	Р	S	Al	Ν
0.3%C	.31	.22	0.76	.003	.003	.030	.001

Pony Mill concept



Existing Hot Mill Line



Experiments





Experimental Routes





Experimental Routes









Effect of deformation temperature



Microstructure







Micro-hardness for different routes



* deformation temperature (PR and BR II) or simulated coiling temperature (BR I)







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Deformation and fracture mechanisms in fine- and ultrafine-grained ferrite/martensite dual-phase steels and the effect of aging

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Abstract

Three ferrite/martensite dual-phase steels varying in the ferrite grain size (12.4, 2.4 and 1.2 μ m) but with the same martensite content (~30 vol.%) were produced by large-strain warm deformation at different deformation temperatures, followed by intercritical annealing. Their mechanical properties were compared, and the response of the ultrafine-grained steel (1.2 μ m) to aging at 170 °C was investigated. The deformation and fracture mechanisms were studied based on microstructure observations using scanning electron microscopy and electron backscatter diffraction. Grain refinement leads to an increase in both yield strength and tensile strength, whereas uniform elongation and total elongation are less affected. This can be partly explained by the increase in the initial strain-hardening rate. Moreover, the stress/strain partitioning characteristics between ferrite and martensite change due to grain refinement, leading to enhanced martensite plasticity and better interface cohesion. Grain refinement further promotes ductile fracture mechanisms, which is a result of the improved fracture toughness of martensite. The aging treatment leads to a strong increase in yield strength and improves the uniform and total elongation. These effects are attributed to dislocation locking due to the formation of Cottrell atmospheres and relaxation of internal stresses, as well as to the reduction in the interstitial carbon content in ferrite and tempering effects in martensite. © 2010 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Ultrafine grains; Dual-phase steel; Aging; Deformation mechanisms; Fracture mechanisms

1. Introduction

Grain refinement of metals is essential as it is the only strengthening mechanism that simultaneously enhances the toughness of a material. In recent years, a variety of methods have been developed to produce ultrafine-grained (UFG) materials with a ferrite grain size of around 1 μ m [1,2]. These methods can be divided into advanced thermomechanical processing (ATMP) routes, which aim at improving conventional processing routes in commercial large-scale rolling mills, and severe plastic deformation (SPD) techniques, which are essentially confined to labora-

tory-scale sample dimensions. The ATMP methods cover deformation-induced ferrite transformation (DIFT) [3], large-strain warm deformation [4], intercritical hot rolling [5], multi-directional rolling [6] and cold-rolling plus annealing of martensitic steel [7]. The most important SPD techniques are equal-channel angular pressing (ECAP) [8], accumulative roll bonding [9] and highpressure torsion [10].

It was consistently found that yield strength and tensile strength are drastically increased due to grain refinement, whereas uniform and total elongation are decreased. Also, Lüders straining becomes more pronounced. Furthermore, UFG steels exhibit a very low strain-hardening rate [11], which marks the main limitation with respect to commercial applications. In terms of toughness, a significant reduction in the ductile-to-brittle transition temperature

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has been repeatedly reported [12,13]. For these reasons, it is of ongoing interest to overcome the restricted ductility of UFG materials without sacrificing strength and toughness. Among the possible ways to restore the strain hardenability of UFG materials are the fabrication of a bimodal grain size distribution [14] and the introduction of nanosized cementite particles into the microstructure [6]. Another aspect that might improve the applicability of UFG materials is their ability to show superplasticity [15]. Shin and Park [16] showed that replacing cementite as a second phase by martensite through an intercritical annealing treatment leads to a substantial increase in strain-hardening rate, causing a higher ultimate tensile strength with only slightly reduced uniform and total elongation. In this way, a UFG ferrite/martensite dual-phase (DP) steel was designed which shows superior mechanical properties compared to coarser grained conventional DP steels. The term "dual-phase steel" refers to a group of steels consisting of a soft ferrite matrix and 3-30 vol.% of hard martensite islands. These steels are widely used for automotive applications. DP steels have a number of unique properties, which include a low elastic limit, high initial strain-hardening rate, continuous yielding, high tensile strength and high uniform and total elongation. Moreover, DP steels exhibit a bake-hardening (BH) effect, i.e. the yield strength increases upon aging at paint-baking temperatures (~170 °C) after forming, giving rise to improved dent and crush resistance. The austenite-to-martensite phase transformation bears the main influence on the mechanical properties of dual-phase steels [17,18]. This phase transformation involves a volume expansion of 2-4% [19], causing an elastically and plastically deformed zone in the ferrite adjacent to martensite [20]. The deformed zone contains a high number of unpinned dislocations [21], giving rise to dislocation heterogeneities in the ferrite. The low elastic limit is thus suggested to be generated by the combined effects of the present elastic stresses that facilitate plastic flow and the additional dislocation, which is assumed to be partly mobile during early stages of yielding [22]. Dislocation-dislocation interactions, dislocation pileups at ferrite/martensite interfaces and the corresponding long-range elastic back stresses contribute to rapid strain hardening.

Previous studies on grain refinement in DP steels consistently revealed that, unlike in other metallic materials, the increases in yield strength and tensile strength are not counteracted by a significant reduction in the uniform and total elongation [23–28]. This can partly be explained by the enhanced strain-hardening rate due to grain refinement as a result of the higher number of geometrically necessary dislocations (GNDs) along the ferrite–martensite boundaries [25]. Ultrafine-grained DP steels have been produced by applying a two-step processing route consisting of (1) a deformation treatment to produce UFG ferrite and finely dispersed cementite or pearlite and (2) a short intercritical annealing in the ferrite/austenite two-phase field followed by quenching to transform all austenite to martensite. Grain refinement in step (1) was achieved by ECAP [27], cold rolling [28], cold swaging [24] and large-strain warm deformation [29]. A single-pass processing route based on DIFT was proposed by Mukherjee et al. [26,30].

As the number of investigations on this topic is very limited, more research is required to understand the mechanical response of DP steels to ferrite grain sizes close to or below 1 µm. Furthermore, as the microstructures described in the previous studies often differ in the martensite volume fraction, it has not been possible to interpret the grain size effect on the mechanical properties independently so far. Therefore, in this study we compare the deformation and fracture mechanisms of a coarse-grained (CG), a finegrained (FG) and a UFG-DP steel, having about the same martensite volume fractions. In addition, the aging (BH) response of the UFG-DP steel is investigated, which has not been addressed so far. In conjunction with the mechanical data, the microstructure evolution during tensile straining and the fracture mechanisms are studied by using scanning electron microscopy (SEM) and high-resolution electron backscatter diffraction (EBSD). Furthermore, slip-band evolution during deformation was investigated by performing tensile tests which were interrupted at strain levels between 1% and 4%.

2. Experimental procedures

2.1. Materials processing

A plain carbon manganese steel of composition (in wt.%) 0.17 C, 1.49 Mn, 0.22 Si, 0.033 Al, 0.0033 N, 0.0017 P and 0.0031 S was produced by vacuum induction melting. A lean composition was selected in order to show that a stable ferrite grain size of around 1 µm can be achieved via thermomechanical processing without microalloving. Carbon enhances both grain refinement and grain size stability [31]. At the same time, the carbon content has to be low enough to ensure good weldability, which is fulfilled for carbon contents below 0.2%. Mn was also shown to be highly beneficial for the grain refinement process [32]. Furthermore, it increases the hardenability and lowers the Ar₃ temperature [29]. However, too high a Mn content will promote segregation and undesired banded microstructures. Samples $(50 \times 40 \times 60 \text{ mm}^3)$ for thermomechanical processing were machined directly from the cast ingot. The thermomechanical processing was realized by use of a large-scale 2.5 MN hot deformation simulator [4,33,34]. This computer-controlled servohydraulic press allows the simulation of industrial hot-rolling processing routes by performing multi-step flat compression tests. The processing schedules to obtain three different grain sizes are outlined in Fig. 1.

The first step is identical for all processing schedules. It consists of 3 min of reaustenitization at 912 °C and a single-pass deformation at 860 °C applying a logarithmic strain of $\varepsilon = 0.3$ at a strain rate of 10 s⁻¹ above the recrystallization temperature. In the CG route (Fig. 1a),



Fig. 1. Thermomechanical processing routes to produce different grain sizes in a hot deformation simulator. All treatments are followed by an intercritical annealing at 730 °C for 3 min and subsequent quenching to obtain the final ferrite/martensite dual-phase microstructure. Ar₃: non-equilibrium transformation start temperature; P_{f} : pearlite transformation finish temperature; ε : logarithmic strain.

the sample is then air cooled to room temperature to obtain a conventional ferrite-pearlite starting microstructure. Grain refinement is achieved by subsequent large-strain warm deformation at 700 °C (the FG route, Fig. 1b) and at 550 °C (the UFG route, Fig. 1c), respectively. A total strain of 1.6 is realized by a four-step flat compression series, each step exerting a strain of 0.4 at a strain rate of 10 s^{-1} . The deformation temperature controls the degree of grain refinement. At 700 °C, a fine polygonal ferrite matrix with globular cementite and fine pearlite colonies is formed. At 550 °C, continuous grain subdivision and pronounced recovery lead to slightly elongated ferrite grains with a grain size of around 1 μ m [4]. The cementite lamellae of the pearlite colonies undergo continuous fragmentation and spheroidization. This results in a fine distribution of spheroidized cementite particles which mainly cover the ferrite grain boundaries. After warm deformation, specimens were annealed for 2 h at the respective deformation temperature to simulate coiling at elevated temperatures, and then air cooled to room temperature.

To obtain the final ferrite/martensite dual-phase microstructure, all specimens were subjected to intercritical annealing in the ferrite/austenite two-phase region followed by quenching in order to transform all austenite into martensite. This treatment was carried out in a salt bath furnace. The temperature was controlled electronically and held constant at 730 °C. The reheating time was 2 min, as was predetermined using a thermocouple embedded in a test specimen. Samples were annealed for 3 min (including reheating time) in the salt bath, before being quenched in water. The intercritical annealing parameters were established by performing dilatometer tests [29].

Three of the UFG samples were additionally aged at 170 °C for 20 min in a vacuum furnace to simulate the BH process, which is active during paint baking in automotive manufacturing. Generally, the increase in yield strength after prestraining between 2% and 12% plus aging at ~170 °C for 20–30 min is determined to assess the

bake-hardenability of a steel grade. Here, only the BH_0 (no prestrain) value for the UFG-DP steel is reported.

2.2. Microstructure characterization

Samples for SEM and EBSD were prepared by standard mechanical grinding and polishing procedures, finishing with 3 min colloidal silica polishing. To reveal the microstructure for SEM observations, the samples were additionally etched in 1% Nital for 3 s. For high-resolution EBSD measurements, the sample surface has to be extremely clean and free of roughness and deformation in order to obtain high-quality Kikuchi patterns. Therefore, they were electropolished using Struers electrolyte A2 at room temperature (voltage: 30 V; flow rate: 12 s^{-1} ; polishing time: 10 s).

The martensite volume fraction and the ferrite grain size were determined on the basis of three SEM micrographs taken at a magnification of $\times 3000$ for the UFG and FG steel and of $\times 500$ for the CG steel. A point-counting method was used to determine the second phase fraction. As it is not possible to differentiate between martensite and austenite on etched specimens in the scanning electron microscope, the second phase fraction was determined as the fraction of martensite plus retained austenite. The retained austenite volume fraction was determined to range between 1 and 3 vol.% based on EBSD measurements. The ferrite mean linear intercept length was determined both in the compression direction and in the rolling direction. The average value determines the ferrite grain size.

EBSD maps were taken on a JEOL JSM 6500F highresolution, high-intensity scanning electron microscope equipped with a field emission gun (FEG SEM) and for energy-dispersive X-ray analysis. The small beam diameter and its high brightness yield high-contrast Kikuchi patterns with a large signal to noise ratio. In this way, information about small orientation deviations can be obtained, even in deformed areas with high dislocation densities like phase or grain boundaries [35,36]. A high-speed DigiView CCD camera was used for pattern acquisition. Data was recorded and analyzed using the EDAX/TSL OIM software package [37]. The lateral resolution of orientation microscopy using the FEG SEM was shown to be of the order of a few tens of nanometers, depending on the material investigated and the microscope, camera and software parameters used [37]. By choosing the highest possible image resolution for pattern acquisition and by optimizing the parameters of the Hough transformation, an angular resolution of about 0.3° can be obtained [37,38]. The step size was 50 nm for the UFG and FG steels and 200 nm for the CG steel. An acceleration voltage of 15 kV and an emission current of around 80 µA were used for all scans. Martensite was indexed as body-centered cubic ferrite. Due to its higher dislocation density, it can be easily distinguished from ferrite by its significantly lower image quality (IQ) and confidence index. In the present work, the minimum misorientation angle that defines a grain boundary was set to 2°. Misorientation angles between 2° and 15° are called low-angle grain boundaries (LAGBs). Misorientation angles larger than 15° are designated high-angle grain boundaries.

Slip-band evolution was studied on the basis of interrupted tensile tests using flat specimens $(5 \times 60 \times 3 \text{ mm}^3)$. Sections containing rolling and normal directions were prepared by mechanical grinding and polishing. Two microhardness indents were placed within the center region of the specimen as a reference to measure the plastic strain after each tensile step. Micrographs and EBSD maps were taken within this distance. The scan position was easily recognized after each straining step due to the slight surface contamination.

2.3. Tensile testing

Cylindrical tensile test specimens with a diameter of 4 mm and a gage length of 20 mm were machined according to the German Industry Norm DIN 50125-B. Tensile tests were conducted at room temperature with a constant cross-head speed of 0.5 mm min^{-1^{-1}} and an initial strain rate of $0.5 \times 10^{-3} \text{ s}^{-1}$. Due to the continuous yielding behavior of the DP steels, the yield strength is given as the 0.2% offset yield strength. In the case of the UFG as-received material, the lower yield strength is reported. The uniform elongation was determined as the strain at which the true strain equals the strain-hardening rate (Considère criterion). The strain-hardening exponent, n, was determined as an approximation to the Hollomon equation $(\sigma_t = k\varepsilon_t^n)$ where σ_t is the true stress, ε_t is the true strain and k is an empirical constant) between 2% and uniform elongation for the DP steels and between the onset of continuous yielding and uniform elongation for the UFG as-received material. The reduction in area was determined by measuring the area of the fracture surface related to the initial cross section.

3. Results

3.1. Microstructures

The microstructure obtained after hot deformation and air cooling followed by intercritical annealing (CG route) consists of a ferrite matrix with a grain size of $12.4 \,\mu\text{m}$ and $31.3 \,\text{vol.\%}$ martensite (Table 1), the latter occurring partly as isolated islands, partly as aligned bands.

By applying multi-pass warm deformation at 700 °C (FG route) and at 550 °C (UFG route) between hot deformation and intercritical annealing, the ferrite grain size is reduced to 2.4 and 1.2 μ m, respectively. The martensite fraction is 30.1 vol.% in the FG steel and 29.8 vol.% in the UFG steel. Exemplary micrographs are shown in Fig. 2. Note that the magnification is the same in all images. Upon BH, the microstructure does not change visibly in the scanning electron microscope, the ferrite grain size being 1.2 μ m and the martensite volume fraction 28.2%.

As neither the chemical composition nor the intercritical annealing temperature or holding time was changed, all three steels contain similar martensite fractions with presumably similar martensite carbon contents. Using a mass balance calculation, the martensite carbon content C_m can be estimated from the equation:

$$C_m = \frac{C_c - C_f (1 - f_m)}{f_m}$$
(1)

where C_c is the carbon content of the composite, C_f is the carbon content of ferrite and f_m is the martensite volume fraction. The ferrite carbon content was estimated using thermo-calc [39]. It was assumed that, upon water quenching, the ferrite keeps the carbon content that is present at the temperature at which the austenite fraction is 30 vol.%. Thus, ferrite is supersaturated in carbon, the carbon content being 0.01 wt.%. Inserting this value in Eq. (1) yields a martensite carbon content of 0.54 wt.%, assuming that no cementite is present in the microstructure.

Other authors conducting similar investigations [24,27] found that phase transformation kinetics is enhanced upon grain refinement. Hence, they report a higher martensite volume fraction in their UFG materials after the same intercritical annealing treatment. The reason why the martensite volume fraction is nearly the same for all grain sizes in the present case is probably the different processing route applied. Due to the pronounced recovery during large-strain warm deformation, the stored energy in the initial microstructure might be lower than in the materials processed by ECAP or cold swaging. Hence, the driving force for phase transformation is not profoundly enhanced in the present case. This leads to the advantageous situation that in this study the differences in the mechanical properties can be solely attributed to the different grain size and these effects are not overlaid by differences in martensite volume fraction. However, it will be shown in the

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Microstructure parameters obtained from SEM micrographs a	nd tensile tests presented as average	e value of three tensile speciment	s for each material.

Steel	MVF (%)	$d_f(\mu m)$	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	RA (%)	Yield ratio	<i>n</i> -value
CG	31.3	12.4	445 ± 17	870 ± 25	7.2 ± 0.7	7.7 ± 1.0	13.0 ± 4.4	0.51 ± 0.01	0.21 ± 0.01
FG	30.1	2.4	483 ± 7	964 ± 4	7.4 ± 0.4	8.9 ± 0.8	18.7 ± 2.6	0.50 ± 0.01	0.18 ± 0.01
UFG	29.8	1.2	525 ± 8	1037 ± 15	7.1 ± 0.5	7.3 ± 0.4	15.3 ± 4.0	0.51 ± 0.01	0.18 ± 0.01
UFG-BH	28.2	1.2	619 ± 6	1005 ± 5	8.6 ± 0.2	11.4 ± 1.5	37.5 ± 0.9	0.62 ± 0.003	0.16 ± 0.004
UFG as-received	_	0.8	578 ± 12	633 ± 6	7.3 ± 0.1	13.3 ± 1.2	61.9 ± 4.6	0.91 ± 0.03	0.06 ± 0.001

Standard deviations are given in brackets. MVF: martensite volume fraction; d_j : ferrite grain size (mean linear intercept length); YS: 0.2% offset yield strength; UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation; RA: reduction in area.



Fig. 2. Microstructures of the (a) CG material (12.4 μ m ferrite grain size), (b) FG material (2.4 μ m ferrite grain size) and (c) UFG material (1.2 μ m ferrite grain size) produced by the processing routes illustrated in Fig. 1 plus intercritical annealing for 3 min at 730 °C in a salt bath, followed by water quenching. As the microstructure does not change visibly upon aging, this microstructure is not shown here.

following that the martensite distribution does have an influence on the mechanical behavior.

3.2. Tensile properties

Fig. 3 shows the engineering stress-strain curves of the CG, FG, UFG and UFG-BH steel. For each material, the result of only one of the three tensile tests is shown, because the variations within each series are rather small (see the standard deviations in Table 1). The CG, FG



Fig. 3. Exemplary engineering stress–strain curves of the steels with CG, FG and UFG ferrite matrix and of the aged (BH) UFG steel. The UFG ferrite/cementite starting material is also shown. Initial strain rate: $0.5 \times 10^{-3} \text{ s}^{-1}$. DP refers to the term "dual-phase".

and UFG steels show the typical characteristics of asquenched ferrite/martensite dual-phase steels: low elastic limit, absence of a distinct yield point, continuous yielding and high initial strain-hardening rate. With decreasing grain size, the yield strength and the tensile strength are increased remarkably whereas uniform elongation and total elongation are only slightly affected. The BH process affects the mechanical properties in two ways: first, it promotes the reoccurrence of a yield point (though the transition between elastic and plastic deformation is still rather smooth); and second, it enhances the ductility considerably in terms of uniform elongation, total elongation and reduction in area. The tensile strength is slightly reduced due to BH. For comparison, the stress-strain curve of the asreceived UFG material is also shown. While the yield strength is slightly higher than in the UFG-DP steel, the strain-hardening rate is much lower, resulting in a relatively low tensile strength. The onset of plastic deformation is characterized by pronounced Lüders straining. The uniform elongation is the same as in the UFG-DP steel, but lower than in the bake-hardened UFG-DP steel. The total elongation is higher than in the other investigated steels; this is ascribed to the large post-uniform plasticity.

Table 1 lists the average values of each steel obtained from three separate tensile tests. The yield strength (0.2% offset yield strength) and tensile strength increase linearly with the inverse square root of the ferrite grain size, i.e. the Hall–Petch relation is obeyed. The grain size dependence (Hall–Petch slope) is 4.0 MPa/ $d^{-1/2}$ for the yield strength (with *d* being the grain diameter in mm), and 8.39 MPa/ $d^{-1/2}$ for the tensile strength. As yield and tensile strength are increased by nearly the same factor due to grain refinement, the yield ratio is nearly constant. The bake-hardened UFG steel does not coincide with these trends. The 0.2% offset yield strength is increased by 94 MPa compared to the as-quenched UFG steel and the tensile strength is decreased by 32 MPa, resulting in a change in the yield ratio from 0.51 to 0.62. As the yield point after BH is more pronounced yet still not sharp, it is reasonable to specify the 0.2% offset yield strength rather than the lower yield strength.

The effect of grain refinement on ductility is more complex than its effect on strength. The uniform elongation (Table 1) is nearly constant around 7% for the CG, FG and UFG steels. Bake-hardening increases uniform elongation to around 8.6%. Regarding only the as-quenched materials, both total elongation and reduction in area are highest in the FG steel. The UFG steel has a lower total elongation than the CG steel but a higher reduction in area. However, the differences are rather small. The BH treatment improves the total elongation by 2.5% and – more significantly – the reduction in area by 22.2%.

The analysis of the strain-hardening rate (Fig. 4) reveals that the initial strain-hardening rate is increased by grain refinement but is nearly the same for the FG and UFG steels. At higher strain levels, the two curves converge with the curve of the CG steel, i.e. the effect of grain refinement continuously decreases. The bake-hardened steel starts with lower strain-hardening rates in the range of the CG steel (for $\varepsilon_t > 0.01$). The curve shows a minimum in the initial stage due to the more pronounced yield point. At higher strain levels, it intersects the other curves, achieving a higher uniform strain. The *n*-value, calculated at strain levels between 2% and uniform elongation (Table 1), drops off slightly from 0.21 for the CG steel to 0.18 for the FG and UFG steels and becomes 0.16 for the bake-hardened steel. The *n*-value of all DP steels is considerably larger than in the UFG ferrite/cementite starting material.



Fig. 4. Strain-hardening rate as a function of true strain. Grain refinement increases the initial strain-hardening rate, whereas BH decreases it. CG, FG, UFG and UFG-BH are as defined in the text.

Fig. 5 shows the tensile specimens after failure. The degree of necking (post-uniform elongation) increases with decreasing grain size and is strongest for the bake-hardened specimens. The micrographs reveal the respective fracture modes of the steels. In the case of the CG steel, it is mainly brittle, which is documented by well-defined facets and cleavage steps on these facets (Fig. 5a). Only some small areas consist of dimples. The latter are located in the martensitic area, whereas the ferrite exhibits cleavage planes, as was observed previously [40,41]. The dominant fracture mode of the FG steel is ductile, although smaller parts of the specimen have undergone brittle fracture (Fig. 5b). The UFG steel shows dimples throughout the specimens, in both the as-quenched (Fig. 5c) and bake-hardened conditions (Fig. 5d). This suggests a failure process of void nucleation and growth, and hence entirely ductile fracture. Some dimples are formed around inclusions.

In order to identify, the preferred void nucleation sites, surfaces perpendicular to the fracture surface were also analyzed. In the CG steel, the main fracture mechanism is martensite cracking. The cracks form mostly in the banded areas perpendicular to the applied tensile strain (Fig. 6a). Most of the cracks stop at the ferrite/martensite interface, but some penetrate into a minor fraction of the adjacent ferrite grain. Martensite fracture was observed at strains as low as 3.4% plastic strain (see below). Void nucleation and growth along ferrite/martensite interfaces occur to a lesser extent within the areas of isolated martensite islands. In the FG and UFG steels, the voids form primarily at ferrite/martensite interfaces and are distributed more homogeneously (Fig. 6b). Martensite cracking takes place less frequently in martensite islands that are of larger than average size and occurs only after necking has started.

3.3. Microstructure evolution during tensile straining

The microstructure evolution during deformation is illustrated for the CG, UFG and UFG-BH steels. The FG steel shows an intermediate deformation behavior. Fig. 7 shows EBSD scans taken perpendicular to the fracture surface of the tensile specimen. In the left column, the IQ maps of areas within the uniform elongation (\sim 7%) are illustrated. The center and right columns show the IQ and inverse pole figure (IPF) maps of areas close to the neck. Martensite is easily identified by its lower IQ and its subdivision into blocks or packets. In all images, the tensile direction (=rolling direction) is horizontal and the normal direction is vertical.

Upon straining to uniform elongation (UE), strain localization in ferrite was observed in the CG steel, as indicated by arrows 1 in Fig. 7a. Locally, a dislocation substructure with vague boundaries has developed (arrow 2). Martensite cracking occurred in some areas (arrow 3). In contrast, the UFG steel shows a less pronounced substructure formation in ferrite. Instead, martensite has undergone considerable deformation and is rotated towards the tensile direction together with the ferrite (arrows in Fig. 7d). The



Fig. 5. Tensile specimen after failure showing the increase in post-uniform elongation with decreasing grain size and the promotion of ductile fracture mechanism (a-c). The aging treatment (BH, BH) enhances this trend (d). CG, FG, UFG and UFG-BH are defined in the text.



Fig. 6. Observation of the planes perpendicular to the fractured tensile specimen surfaces reveals (a) martensite cracking as the main fracture mechanism in the CG specimen and (b) void nucleation and growth in the UFG specimen. Note the different magnification of the images. The tensile direction is horizontal; the normal direction is vertical.

same is true for the UFG-BH steel. Nevertheless, the martensite is even more deformed compared to the non-bake-hardened state, as is revealed by the more elongated shape of the martensite islands (Fig. 7g).

After failure, strain localization and substructure formation of the CG ferrite is intense, particularly close to ferrite/ martensite interfaces. Martensite cracking and interface decohesion are commonplace in this state (arrows in Fig. 7b), while plastic deformation of martensite is not obvious. In the UFG steel (Fig. 7e), it can be easily seen that the martensite is extensively deformed and elongated in the tensile direction. Compared to the CG steel, the deformation substructure in the ferrite is less well developed. Moreover, the distribution of the deformation substructure within the ferrite grains is more homogeneous than in the CG structure. Thus, it seems that strain localization and dynamic recovery are less active in the UFG steel than in the CG steel. On the other hand, the plastic deformation of martensite is more intense. Voids form mainly along the ferrite/martensite interface (arrows in Fig. 7e) and are also elongated in the tensile direction. In the bake-hardened condition, martensite plasticity is



Fig. 7. EBSD maps taken perpendicular to the fracture surface of the CG specimen (a–c), the UFG specimen (d–f) and the UFG-BH specimen (g–i). (a, d, g) IQ maps of areas within the area of UE. IQ maps taken close to the neck (b, e, h) and the respective IPF maps (c, f, i) show the microstructure evolution during straining. The tensile direction is horizontal and equals the rolling direction; the normal direction is vertical.

further enhanced. As indicated by the much higher reduction in area compared to the CG and UFG steels, a subgrain structure is widely developed in the UFG-BH steel.

The IPF maps reflect the different types of strain accommodation in ferrite. The substructure formation in the CG steel results in pronounced orientation gradients which are randomly distributed within the ferrite grains (Fig. 7c), whereas the deformation of the UFG steel results in more planar arrays of crystal orientations in an angle around 40° to the tensile direction (Fig. 7f). The IPF map of the UFG-BH looks similar, yet the subgrain formation is reflected by a finer subdivision of the ferrite grains.

In Fig. 8, typical misorientation profiles of the different ferrite substructures after failure are presented. Their positions are indicated on the respective IQ maps (red¹ lines in Fig. 7b, e and h). From these profiles, it is evident that the UFG-BH sample is the only one which exhibits full development of subgrains that are separated by LAGBs. This is revealed by the stepwise increase in the misorientation angle. The presence of fully developed subgrains indicates that dynamic recovery was active in the necked area of



Fig. 8. Point-to-origin misorientation profiles for the UFG and the UFG-BH samples (lower *x*-axis) compared to CG sample (upper *x*-axis), taken from EBSD maps close to the neck. Subgrain formation is completed only in the UFG-BH sample.

the bake-hardened sample. In the other two specimens, plastic deformation leads to pronounced orientation gradients and to a less extent to well-defined subgrains. This

¹ For interpretation of color in Fig. 7, the reader is referred to the web version of this article.

gives rise to more gradually increasing misorientation profiles. Note the different scaling for the CG sample, which indicates a larger size of the subgrains to be formed. The subgrain size of the UFG-BH steel is around $0.3 \,\mu\text{m}$.

The microstructure evolution during deformation was studied in more detail by interrupted tensile tests. The UFG-BH was excluded from this part of the investigation because the tempered state of the microstructure obviously alters the deformation mechanisms and therefore it is not comparable to the untempered state. Although the EBSD scans revealed important information about the deformation substructure, single slip bands cannot be distinguished. Therefore, the evolution of slip bands was studied on polished samples subjected to different strain levels. The CG ferrite exhibits wavy and strongly intersecting slip bands without preferred orientation after 3.4% plastic strain (Fig. 9a). In contrast, the UFG ferrite basically shows two sets of nearly planar slip bands which are oriented around 40° to the tensile direction, as was observed in the IPF maps (Fig. 9b). This suggests that less pronounced orientation gradients are developed in the UFG steel at this strain than in the CG steel, as was observed in the IPF

maps in Fig. 7. This assumption was further tested by analyzing the evolution of local misorientations at small strains. For this purpose, the kernel average misorientation (KAM) was used which can be directly retrieved from the EBSD data.

The KAM is defined as the average misorientation angle between an EBSD measurement point and all its neighbors at a certain distance. Given that the KAM angle is strongly dependent on the distance selected, the nearest neighbor used for the calculation was determined based on the following considerations: the distance has to be sufficiently small to ensure that detailed information about local misorientation changes is not ignored and that influences stemming from neighboring grains are not taken into account. At the same time, the distance must allow for averaging out scatter due to the spatial resolution limits of EBSD and for performing the calculations with misorientations above the angular EBSD resolution limit [44]. Hence, for both samples, the CG and the UFG material, neighboring points located at a distance of 200 nm spacing were selected. Misorientation angles above 5° were excluded from the calculation. This angle is large enough to still



Fig. 9. The CG steel exhibits wavy slip bands (a), whereas slip-band formation in the UFG sample follows a more planar mode (b). The tensile direction is horizontal. The distribution of the KAM values (calculated using neighboring EBSD points at a distance of 200 nm and up to a maximum deviation angle of 5°) at different strain levels in the same sample area shows that lattice rotations take place early during tensile straining in the CG steel (c), whereas they appear delayed in the UFG specimen (d) due to the more constrained plastic deformation in ferrite. The rolling direction is horizontal; the normal direction is vertical.

include in the analysis the subgrains that evolve during plastic deformation, yet is small enough to exclude neighboring grains from the calculation.

The calculation was done at different strain levels on the same sample area. Only the ferrite fraction was taken into account. The results are shown in Fig. 9c and d. In the CG steel, a uniform shift of the peak KAM value to higher misorientation angles and a gradual increase in the fraction of higher misorientation angles are observed. The flattening of the curve indicates that strain is distributed inhomogeneously throughout the ferrite grains. A larger fraction of high misorientation points to the increasing number of dislocation walls or subgrains. The peak value of the KAM at 0% strain is lower in the UFG steel than in the CG steel due to the larger fraction of dislocations at grain boundaries. Up to 3% plastic strain, the KAM distribution remains unchanged. Only after failure was a considerable decrease in the peak value found, accompanied by an increased fraction of high misorientation angles. Hence, the planar slip mode in the UFG steel correlates with restricted lattice rotations and thus delayed subgrain formation. In contrast, non-planar glide, including the intense lattice rotations and earlier formation of a substructure, prevail in the CG steel.

4. Discussion

For the sake of clarity, the discussion of the deformation and fracture mechanisms will focus on the as-quenched specimens only. The effect of BH on deformation and fracture behavior will be addressed in a separate section.

4.1. Deformation mechanisms

In general, the enhancement of strength due to grain refinement is accompanied by a deterioration of ductility. However, it was shown in previous studies [23–28] that this does not apply to DP steels. Instead, it was found that uniform and total elongation are only slightly affected by decreasing ferrite grain size - as was also observed in the present study. This is partly a consequence of the increase in the initial strain-hardening rate with decreasing grain size [25,27,42,43]. In a recent paper [44], the enhancement of the initial strain-hardening rate was explained in terms of reductions in the martensite island size and ferrite grain size. These effects were suggested to enhance the number of dislocation sources and give rise to rapid dislocation interactions. The higher ferrite/martensite phase boundary fraction results in a larger number of GNDs, which are generated during plastic deformation due to the strain incompatibility of the two phases. Furthermore, the back stresses exerted by (i) martensite islands and (ii) ultrafine ferrite grains below $1 \,\mu\text{m}^3$, which are characterized by a more uniform dislocation distribution than larger grains, contribute to the rapid stress increment. It is assumed that the deformation-induced transformation of small amounts of retained austenite plays a secondary role.

When focusing on low strain levels <2%, it must be stated that the increase in yield strength due to ferrite grain refinement contributes to the high initial strain-hardening rate. Due to the absence of a distinct yield point, it is not possible to clearly distinguish between the effect of grain size on strain-hardening rate and that on yield strength. This becomes clear when looking at the strain-hardening rate of the UFG-BH steel, which is nearly equal to the strain-hardening rate of the CG steel. To establish a consistent comparison of the strain-hardening capacity of UFG ferrite/cementite and UFG ferrite/martensite steels, it is thus necessary to analyze UFG ferrite/martensite steels after strain aging, which exhibit a distinct yield point.

The aspect that shall be treated in more detail in the present study is the plasticity of martensite, which is considerably enhanced due to grain refinement. In general, plastic yielding starts in the soft ferrite, with the hard martensite remaining in the elastic state. During the plastic deformation of the ferrite, stress is transferred to the martensite. Internal stresses at the ferrite/martensite interface are built up due to the plastic strain incompatibility. At this stage, the strain-hardening rate is very high as a consequence of rapid dislocation multiplication and the back stresses resulting from the strain incompatibility. When the transferred stress is large enough to reach the elastic limit of the martensite, it starts to deform plastically. Due to the high initial strain hardening of the martensite, the stress increment in the composite is still very high. Only after the martensite flow curve levels off, its contribution to the strain-hardening rate of the composite declines [45]. From these general considerations, it is clear that martensite plasticity is an important factor controlling the overall deformation behavior of DP steels. Generally, the ability of martensite to deform increases with a decay in hardness, which can be reduced by lowering the carbon content or by applying tempering. In view of a composite, a lower strength difference between martensite and ferrite promotes plastic deformation of martensite [46]. Although some studies have reported that plastic deformation of martensite occurs only after uniform elongation (e.g. [42,47]), considerable martensite deformation at lower strains was shown by in situ SEM studies [48,49] and by analyzing the two-stage deformation behavior derived from the strain-hardening rate [25]. In the present materials, both types of behavior have been observed. The martensite in the CG steel seems to remain in the elastic state even in the necked area of the tensile specimen. On the other hand, the FG and UFG steels clearly show considerable martensite deformation before the onset of necking. According to the model developed by Jiang et al. [25], the increase in the yield strength and the strain-hardening rate of the ferrite matrix due to grain refinement results in rapid stress transfer to martensite. Thus, the yield stress of martensite is reached at lower strains than in coarser-grained microstructures. However, it must be assumed that the yield strength of martensite also increases with grain refinement due to the finer packet size. This, in turn, would delay martensite plastic deformation.

In order to identify the reason for the enhanced martensite plasticity due to grain refinement, one has to consider how stress and strain are partitioned between ferrite and martensite. In this respect, the slip-band evolution reveals important information about plastic strain accommodation. In the CG steel, the slip bands appear wavy (Fig. 9a), whereas in UFG steel a more planar slip-band formation is active (Fig. 9b). Planar and wavy slip modes in DP steels were reported by Tomota [50]. Tomota explains the occurrence of planar slip with the restricted operation of plastic relaxation of strain incompatibility while the wavy slip mode is associated with active plastic relaxation. Plastic relaxation can be realized by (i) additional plastic flow in the softer phase, (ii) onset of plastic flow in the harder phase, (iii) fracture of the harder phase or (iv) decohesion at the interface. In the CG steel, plastic relaxation can take place by strain localization and substructure formation in ferrite and by martensite cracking (Fig. 7a and b). Therefore, slip bands develop in a wavy mode (Fig. 9a). This deformation mechanism is accompanied by intense lattice rotations, which are reflected by the gradual increase in local misorientations (Fig. 9c). The finer the ferrite grain size is, the more difficult the plastic deformation of ferrite becomes due to the restricted formation of dislocation pile-ups. The restricted ferrite plasticity is reflected by the planar slip mode (Fig. 9b) and only a few lattice rotations (Fig. 9d). Due to the nearly spherical shape and the resulting high toughness of the martensite islands, both interface decohesion and cracking are impeded at low strains. At later stages of plastic deformation (more than 3%), plastic relaxation in the UFG steel occurs by the onset of martensite plastic flow and by interface decohesion (Fig. 7e). Hence, the plasticity of martensite is strongly dependent on the plastic constraints in the ferrite. The plastic constraint imposed on UFG ferrite is balanced by the more spherical morphology of UFG martensite compared to the CG martensite, the latter often covering ferrite grain boundaries. At a constant martensite volume fraction, the spherical shape toughens martensite and increases the ductility of the composite. Therefore, the UFG steel shows ductility comparable to that of the CG steel at much higher strength levels due to the advantageous morphology and distribution of martensite and the ability of martensite to deform plastically as a response to the restricted plasticity in ferrite.

It is evident from these observations that stress/strain partitioning, which is essential in this type of composite structure, changes significantly upon grain refinement. In the CG steel, stress/strain partitioning is extensive in the sense that martensite carries the major part of the stress and ferrite carries the major part of the strain. This leads to strain localization in ferrite and to early cracking of martensite. Upon grain refinement, ferrite is strengthened more than martensite, as the yield strength of martensite is mainly a function of its carbon content, which is supposed to be nearly constant in all investigated materials. In particular, ferrite grains below $1 \ \mu m^3$ will act as a load-carrying phase. Using three-dimensional EBSD measurements, these grains were shown to be often entirely prestrained by the volume expansion accompanying the austenite-to-martensite phase transformation [44]. Thus, the hardness difference between ferrite and martensite is presumably decreased due to grain refinement. In this situation, the hard phase (martensite) is forced to undergo plastic deformation at lower strains [45]. Thus, stress and strain are partitioned more evenly between martensite and ferrite in the UFG material. This leads to less severe strain incompatibility at the ferrite/martensite interface, resulting in better interface cohesion, and therefore to delayed void formation and an improved post-uniform elongation (Fig. 5).

More research is needed to fully understand the deformation mechanisms in this UFG composite structure. In this respect, digital image correlation is a suitable technique to quantify the strains accommodated by ferrite and martensite [48,51], and will be applied in the future. Furthermore, the hardness difference between ferrite and martensite in the different materials shall be quantified using nanoindentation.

4.2. Fracture mechanisms

Grain refinement promotes ductile fracture mechanisms (Fig. 5). In the CG material, brittle fracture behavior is favored due to martensite banding, a large martensite island size and unfavorable distribution along ferrite grain boundaries. Voids and cracks are distributed mainly around martensite bands (Fig. 6), where local stresses concentrate. As stress is transferred to martensite during tensile straining of DP steels, the fracture stress in martensite is reached much earlier than in ferrite. Therefore, fracture of martensite is initiated. Martensite cracking is facilitated by (i) the low toughness of the martensite islands and (ii) the presence of former austenite-austenite grain boundaries, which are known to be brittle due to their high susceptibility to segregations [52]. The initiated microcracks impose a high shear stress on the neighboring ferrite, which increases with the martensite effective grain size. Hence, failure of coarse martensite islands leads to cleavage fracture of ferrite [53]. As a consequence, premature martensite cracking controls both tensile strength and uniform elongation in the CG steel.

In the FG and UFG steels, martensite cracking is less frequent as a result of the enhanced martensite plasticity and the better interface cohesion as described above. By deforming, martensite releases part of the local stress concentrations and retards void formation, which results in higher fracture strains. Moreover, it is known that the plastic strain needed for the failure of a particle (or grain) increases with decreasing particle size. This is explained by the smaller number of dislocations piling-up at grain and phase boundaries, which result in lower shear stresses [41]. If martensite failure occurs, the produced cracks are not large enough to initiate cleave fracture of the adjacent ferrite. This is because the length of the crack, which determines the peak stress at the crack tip, is restricted by the grain size. Thus, grain refinement increases the cleavage fracture stress and promotes ductile fracture mechanisms at room temperature.

These findings are similar to the results of Kim and Thomas [54], who found that coarse DP structures fracture predominantly by cleavage, while both fine fibrous and fine globular structures fracture in a ductile manner. They attribute this behavior to the constrained possibility of deformation localization in the fine structures, which reduces the probability of cleavage crack nucleation in ferrite. Indeed, the observed strain localization in the CG material is likely to induce cleavage fracture in ferrite. As less excess strain is generated in UFG ferrite, less excess stress is exerted on the martensite phase. Thus, the more even stress and strain partitioning promotes the void nucleation and growth process that is characteristic of ductile fracture behavior. In the CG steel, more stress is transferred to martensite, leading to premature local fracture of martensite and mainly brittle fracture behavior with nearly absent post-uniform elongation.

4.3. Aging response of the UFG material

The BH (aging) process induces static strain aging phenomena in the ferrite and tempering phenomena in the martensite [55]. Carbon atoms in solid solution form Cottrell atmospheres around dislocations and grain boundaries or precipitate as transition carbides in ferrite and/or martensite [56]. The diffusion of carbon includes a volume decrease in martensite, which reduces the internal stresses in ferrite. The formation of Cottrell atmospheres around dislocations causes (partial) pinning of these dislocations. In addition to the reduction in internal stresses, this leads to the reoccurrence of a more distinct yield point and to an increase in the yield strength. In the present case, no sharp yield point was regenerated after BH, but the 0.2% offset yield strength increases by nearly 100 MPa. The dislocation pinning and release of internal stresses successfully suppressed the early onset of plastic deformation of ferrite. The strengthening effect of carbon precipitation is accompanied by a loss in hardness due to the removal of supersaturated carbon in solid solution. This effect might be the reason for the reduction in tensile strength by 32 MPa after BH. Similar findings were proposed by Tanaka et al. [57], who attribute the decrease in tensile strength to the reduction in the amount of excessively dissolved carbon in ferrite. This was experimentally confirmed by Nakaoka et al. [58]. The combined effects of the increase in yield strength and decrease in tensile strength are assumed to be the reason for the lower strain-hardening rate compared to the non-bake-hardened steel (Fig. 4).

The effect of BH on yield strength is more pronounced than on tensile strength, which is consistent with the findings on CG DP steels [59]. The common explanation is that aging phenomena, including the formation of Cottrell atmospheres around dislocations, mainly affect the onset of yielding by restricting the dislocation motion, thereby increasing the yield strength. As soon as the dislocations tear off the Cottrell atmospheres, yielding and strain hardening are controlled by dislocation motion and multiplication, as well as by the hardness difference between the phases. Hence, aging phenomena play a minor role in the strain range of the tensile strength.

The most remarkable effect of BH is the increase in total elongation and reduction in area. One reason for the increased ductility might be the above-mentioned decrease in ferrite hardness due to a loss of dissolved carbon, as was suggested by Koo and Thomas [60]. The other reason is the decrease in martensite hardness due to tempering effects. Decreasing the hardness of martensite increases the critical strain required for interface decohesion or martensite cracking. As a consequence, extensive deformation of martensite was found in the necked area of the tensile specimen (Fig. 7h). As the martensite plasticity retards the formation of voids, plastic deformation of ferrite can continue. Therefore, a rather homogeneous subgrain structure in the ferrite is formed close to the necking zone of the UFG-BH sample (Figs. 7h and 8). The beneficial effect of tempering on decreasing the strength of martensite and, therefore, promoting its plastic deformation was documented for coarser-grained microstructures by Kang et al. [48] and Mazinani and Poole [61].

5. Conclusions

Three low-carbon dual-phase steels with nearly constant martensite fraction around 30 vol.% martensite and different ferrite grain sizes (1.2, 2.4 and 12.4 μ m) were produced by applying hot deformation and large-strain warm deformation at different deformation temperatures, followed by intercritical annealing. Their deformation and fracture mechanisms were studied based on tensile test data and microstructure observations. The BH response was investigated for the UFG steel. The main conclusions are:

- Grain refinement leads to an increase in both yield strength and tensile strength following a linear relationship of the Hall–Petch type. Uniform elongation and total elongation are hardly affected. The initial strainhardening rate and the reduction in area increase as the grain size decreases.
- The increase in the initial strain-hardening rate due to grain refinement is attributed to early dislocation interactions, the high number of dislocation sources and the back stresses exerted by (i) martensite islands and (ii) very small ferrite grains below $1 \ \mu m^3$.
- Aging at 170 °C (bake-hardening) of the UFG steel leads to a strong increase in yield strength and a small decrease in tensile strength. Ductility is enhanced in terms of uniform and total elongation. Reduction in area is improved by 22% (from 15.3% to 37.5%). These effects are attributed to dislocation locking, relaxation of

internal stresses, reduction in supersaturated interstitial carbon content in ferrite and tempering effects in martensite.

- Grain refinement leads to plastic constraints in the ferrite matrix, which is reflected by homogeneous planar slip-band arrays. Strain localization in CG ferrite is accomplished by wavy slip bands. The wavy slip mode leads to pronounced lattice rotations and early formation of a substructure in CG ferrite, whereas the planar slip mode provokes less lattice rotations. The plastic constraints in FG and UFG ferrite force martensite to deform plastically earlier during tensile straining, whereas strain localization and subgrain formation are impeded.
- The increase in strength at improved ductility due to grain refinement is attributed to the combined effect of strengthened ferrite and enhanced toughness of martensite. This leads to less severe stress/strain partitioning and better interface cohesion.
- Grain refinement promotes ductile fracture mechanisms. Besides the beneficial effects of less excess strain in ferrite and less excess stress in martensite, the formation of martensite cracks and cleavage fracture in ferrite is suppressed in the FG and UFG steels due to the small size, the more homogeneous distribution and the more spherical shape of martensite islands.

References

- [1] Howe AA, et al. ECSC contract 7210.PR/167. Luxembourg: EC; 2000.
- [2] Song R, Ponge D, Raabe D, Speer JG, Matlock DK. Mater Sci Eng A 2006;441:1.
- [3] Hodgson PD, Hickson MR, Gibbs RK. Mater Sci Forum 1998;284– 302:63.
- [4] Song R, Ponge D, Raabe D, Kaspar R. Acta Mater 2005;53:845.
- [5] Yada H, Matsumura Y, Nakajima K. US patent 4466,842. Tokyo, Japan: Nippon Steel Corporation; 1984.
- [6] Ohmori A, Torizuka S, Nagai K. ISIJ Int 2004;44:1063.
- [7] Ueji R, Tsuji N, Minamino Y, Koizumi Y. Acta Mater 2002;50:4177.
- [8] Valiev RZ, Langdon TG. Prog Mater Sci 2006;51:881.
- [9] Saito Y, Utsunomiya H, Tsuji N, Sakai T. Acta Mater 1999;47:579.
- [10] Ivanisenko Y, Lojkowski W, Valiev RZ, Fecht HJ. Acta Mater 2003;51:5555.
- [11] Park KT, Shin DH. Metall Mater Trans A 2002;33:705.
- [12] Hanamura T, Yin F, Nagai K. ISIJ Int 2004;44:610.
- [13] Song R, Ponge D, Raabe D. Acta Mater 2005;53:4881.
- [14] Azizi-Alizamini H, Militzer M, Poole WJ. Scr Mater 2007;57:1065.
- [15] Zhang ZL, Liu YN, Zhu JW, Guang YU. Mater Sci Forum 2007;551–552:199.
- [16] Shin DH, Park K-T. Mat Sci Eng A 2005;410-411:299.
- [17] Hayami S, Furukawa T. In: Korchynsky M, editor. Microalloying 75 proceedings of an international symposium on high-strength, lowalloy steels. New York: Union Carbide Corporation; 1977. p. 311.
- [18] Speich GR, Miller RL. In: Kot RA, Morris JW, editors. Structure and properties of dual-phase steels. New York: The Metallurgical Society of AIME; 1979. p. 145.
- [19] Moyer JM, Ansell GS. Metall Trans A 1975;6:1785.
- [20] Bourell DL, Rizk A. Acta Metall 1983;31:609.

- [21] Davies RG. Metall Trans A 1978;9:41.
- [22] Rigsbee JM, Vander Arend PJ. In: Davenport AD, editor. Formable HSLA and dual-phase steels. New York: The Metallurgical Society of AIME; 1979. p. 58.
- [23] Chang PH, Preban AG. Acta Metall 1985;33:897.
- [24] Delincé M, Brechet Y, Embury JD, Geers MGD, Jacques PJ, Pardoen T. Acta Mater 2007;55:2337.
- [25] Jiang ZH, Guan ZZ, Lian JS. Mater Sci Eng A 1995;190:55.
- [26] Mukherjee K, Hazra SS, Militzer M. Metall Mater Trans A 2009;40A:2145.
- [27] Son YI, Lee YK, Park KT, Lee CS, Shin DH. Acta Mater 2005;53:3125.
- [28] Tsipouridis P, Werner E, Krempaszky C, Tragl E. Steel Res Int 2006;77:654.
- [29] Calcagnotto M, Ponge D, Raabe D. ISIJ Int 2008;48:1096.
- [30] Mukherjee K, Hazra S, Petkov P, Militzer M. Mater Manuf Process 2007;22:511.
- [31] Song R. Microstructure and mechanical properties of UFG C-Mn steels. Aachen: Shaker Verlag; 2005.
- [32] Song R, Ponge D, Raabe D. ISIJ Int 2005;45:1721.
- [33] Kaspar R, Pawelski O. Materialprüfung 1989;31:14.
- [34] Pawelski O, Kaspar R. Materialprüfung 1988;30:357.
- [35] Humphreys FJ. J Mater Sci 2001;36:3833.
- [36] Schwartz AJ, Kumar M, Adams BL, Field DP. Electron backscatter diffraction in materials science. New York: Springer; 2009.
- [37] Dingley D. J Microsc-Oxford 2004;213:214.
- [38] Bate PS, Knutsen RD, Brough I, Humphreys FJ. J Microsc-Oxford 2005;220:36.
- [39] Jansson B, Schalin M, Selleby M, Sundman B. In: Bale CW, Irons GA, editors. Computer software in chemical and extractive metallurgy. Quebec: The Metallurgical Society of CIM; 1993. p. 57.
- [40] Cai XL, Feng J, Owen WS. Metall Trans A 1985;16:1405.
- [41] He XJ, Terao N, Berghezan A. Met Sci 1984;18:367.
- [42] Balliger NK, Gladman T. Met Sci 1981;15:95.
- [43] Calcagnotto M, Ponge D, Raabe D. Mater Sci Eng A 2010;527:7832–40. doi:10.1016/j.msea.2010.08.062.
- [44] Calcagnotto M, Ponge D, Demir E, Raabe D. Mater Sci Eng A 2010;527:2738.
- [45] Fischmeister H, Karlsson B. Z Metallkd 1977;68:311.
- [46] Lian J, Jiang Z, Liu J. Mater Sci Eng A 1991;147:55.
- [47] Marder AR. Metall Trans A 1982;13:85.
- [48] Kang JD, Ososkov Y, Embury JD, Wilkinson DS. Scr Mater 2007;56:999.
- [49] Su YL, Gurland J. Mater Sci Eng 1987;95:151.
- [50] Tomota Y. Mater Sci Technol 1987;3:415.
- [51] Ghadbeigi H, Pinna C, Celotto S, Yates JR. Mater Sci Eng A 2010;527:5026.
- [52] Becker J, Hornbogen E, Stratmann P. Z Metallkd 1980;71:27.
- [53] Uggowitzer P, Stüwe HP. Mater Sci Eng 1982;55:181.
- [54] Kim NJ, Thomas G. Metall Trans A 1981;12:483.
- [55] Waterschoot T, Verbeken K, De Cooman BC. ISIJ Int 2006;46:138.
- [56] Speich GR, Miller AB. In: Kot RA, Bramfitt BL, editors. Fundamentals of dual-phase steels. New York: The Metallurgical Society of AIME; 1981. p. 279.
- [57] Tanaka T, Nishida M, Hashiguchi K, Kato T. In: Kot RA, Morris JW, editors. Structure and properties of dual-phase steels. New York: The Metallurgical Society of AIME; 1979. p. 221.
- [58] Nakaoka K, Hosoya Y, Ohmura M, Nishimoto A. In: Kot RA, Morris JW, editors. Structure and properties of dual-phase steels. New York: The Metallurgical Society of AIME; 1979. p. 330.
- [59] Davies RG. Metall Trans A 1979;10:1549.
- [60] Koo JY, Thomas G. In: Davenport AD, editor. Formable HSLA and dual-phase steels. New York: The Metallurgical Society of AIME; 1979. p. 40.
- [61] Mazinani M, Poole WJ. Metall Mater Trans A 2007;38A:328.



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Development of microstructure and texture of medium carbon steel during heavy warm deformation

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Abstract

The microstructure and texture development of a medium-carbon steel (0.36% C) during heavy warm deformation (HWD) was studied using scanning electron microscopy and electron back scattering diffraction. The spheroidization of pearlite is accelerated due to the HWD, which leads to the formation of completely spheroidized cementite already after the deformation and coiling at 873 K (600 °C). The homogeneity of the cementite distribution depends on the cooling rate and the coiling temperature. The cooling rate of about 10 K/s (ferrite–pearlite prior to HWD) and deformation/coiling at 943–973 K (670–700 °C) lead to a homogeneous cementite distribution with a cementite particle size of less than 1 µm. The ferrite softening can be attributed to continuous recrystallization. Even up to fairly high deformation/coiling temperatures of 983 K (710 °C) the texture consists of typical deformation components. During the continuous recrystallization the amount of high angle grain boundaries can increase up to 70% with a ferrite grain size of 1–3 µm. An increase of the cooling rate up to 20 K/s (ferrite–pearlite–bainite prior to HWD) deteriorates the homogeneity of the cementite distribution and the softening of ferrite in the final microstructure. © 2004 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

Keywords: Heavy warm deformation; Ferritic-pearlitic steel; Continuous recrystallization; Texture; EBSD

1. Introduction

After conventional hot rolling of medium carbon steel, a lamellar pearlite is formed during γ - α transformation. The lamellar morphology of pearlite leads to mechanical properties unsuitable for a further cold treatment or for application in highly demanding components. The globular morphology of cementite provides some benefits such as high toughness, good cold formability and machinability. For such purposes the cold strip must either undergo a long annealing treatment to obtain higher cold formability or it must be quenched with a subsequent tempering for a good combination of strength and toughness.

The use of a heavy warm deformation (HWD), performed below the γ - α transformation-temperature accelerates essentially the spheroidization of pearlite.

The rate of this process is accelerated by a factor of 10^4 compared to annealing without deformation [1,2]. But the spheroidized cementite itself cannot provide good mechanical properties. Other microstructural features like a homogeneous cementite distribution or ferrite condition, as well as the size of the cementite particles or the ferrite grains, influence the final mechanical properties. Therefore, a better understanding of the microstructure evolution during HWD is important for a successful introduction of such processing into the industrial production.

2. Material and experimental technique

A ferritic–pearlitic steel with a following composition in mass % was studied: 0.36% C, 0.53% Mn, 0.22% Si, 0.011% P and 0.002% S. The axially symmetric compression of samples with initial size $18 \times 18 \times 30$ mm³ and plane strain compression of samples with an initial thickness of 60 mm, width of 50 mm and length of 45

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mm, cut from an industrial slab, were carried out on the hot deformation simulator of the Max-Planck-Institute [3] with a strain rate of 10 s^{-1} . This servohydraulic press is capable of conducting large-scale thermomechanical processes by performing multi-step hot compression tests as a realistic approximation of industry-scale hot forming operations.

After the austenite deformation (true strain of 0.3) at 1173 K (900 °C), the samples were cooled with various cooling rates between 2 and 20 K/s to obtain the γ - α transformation. The HWD was carried out at 873–983 K (600–710 °C) using a multi-pass mode (4 passes × 0.4 strain, interpass time around 0.5 s) with a subsequent coiling simulation at 873–983 K (600–710 °C) for 2 h.

The deformation-dilatometry technique was used for the establishment of CCT diagrams after the deformation in austenite. The microstructure investigation was carried out using scanning electron microscopy. The condition of the ferrite was additionally studied by the electron back scattering diffraction (EBSD).

3. Results and discussion

3.1. CCT diagram

The continuous cooling transformation diagram (Fig. 1) was established after an austenite deformation at 1173 K (900 °C). This temperature was determined by pre-tests to provide fine recrystallized austenite grains as an initial microstructure before the transformation.

The main differences between the microstructures produced with different cooling rates (initial microstructures for the subsequent HWD) were the amount of proeutectoid ferrite, the presence of bainite and the



Fig. 1. CCT diagram after austenite deformation.

thickness of pearlite lamellae. After slow cooling (2 K/s) the microstructure contains coarse lamellar pearlite with a thick network of proeutectoid ferrite. After cooling with a faster cooling rate of 8 K/s, the ferrite network is thinner and the lamellae are finer. After higher cooling rates (30 K/s), the microstructure contains fine lamellar pearlite, bainite and martensite with only a small amount of ferrite.

3.2. Spheroidization of pearlite

Pearlite colonies with a different orientation after HWD with subsequent cooling at a low rate, 2 K/s, and deformation/coiling temperatures of 873 K (600 °C) are shown in Fig. 2(a) and (b). The formation of spheroidized cementite particles along the former pearlite lamellae can be seen. According to Chattopadhyay and Sellars [4], an excess of vacancies, which formed during the deformation, promotes carbon diffusion, especially near lamellae kinks, which are characteristic of severely deformed pearlite [2,5]. An important factor for the acceleration of the spheroidization process can be a local difference between the equilibrium carbon concentrations in ferrite near the surface of a deformed lamella with different curvature radii, according to the Gibbs-Thompson equation. As reported in [6,7], the equilibrium carbon concentration in ferrite in the vicinity of the lamella with a small curvature radius is higher compared to that of larger one. After a heavy deformation of pearlite the numerous kinks of the lamellae occur with small radii and so with the equilibrium carbon concentration in ferrite near kinks essential higher as compared to that close to the flat parts of lamellae. Together with the high defect density the carbon diffusion leads to a rapid dissolution of lamellae kinks and a simultaneous deposition of carbon in the flat cementite lamella.

The fracture of lamellae (cf. Fig. 2(b), single arrow) in the pearlite colonies with lamellae oriented perpendicular to the rolling direction can also accelerate the spheroidization. Fragments of the former lamellae located at a prior austenite grain boundary (cf. Fig. 2(b), double arrow) can easily be formed because of an accelerated diffusion along the boundary, which leads to a faster coarsening of these cementite fragments.

For the case of a heavy deformation the substructure of the pearlite lamellae can also exhibit an essential effect on the spheroidization process. As reported in [7], the interface adjacent to the subboundary in the cementite lamella with a large local curvature and the surrounding ferrite provokes a quick carbon dissolution that leads to a local lamella division (double arrow in Fig. 2(c)).

The start of the spheroidization of the cementite lamellae in the vicinity of ferrite subboundaries, as reported in [8], was also observed (cf. Fig. 2(c), single arrow). In this case the subboundary facilitates the deposition of carbon in contact place with the lamella. But



Fig. 2. Spheroidization of pearlite: (a) cementite particles along the former lamellae; (b) fracture of lamellae (single arrow) and the lamella fragments at a prior austenite grain boundary (double arrow); (c) enhancing effect of ferrite boundary (single arrow) and cementite subboundaries (double arrow) on start of spheroidization and (d) former pearlite colony (double arrow shows the spheroidization of kinked lamella).

this mechanism does not seem to have a first-order effect for a heavily warm deformed steel. The present EBSD study has shown that the processes of spheroidization and redistribution of cementite take place already in a deformed ferrite, when subboundaries do not yet exist.

The appearance of a typical former pearlite colony at the end of the spheroidization process is shown in Fig. 2(d). Along the former lamellae the ferrite boundaries with the cementite chains can be seen. The ferrite between the chains is elongated and seems to be deformed. Some cementite particles have a prolonged form. The spheroidization of the last kinked lamella can be also observed (cf. Fig. 2(d), double arrow).

3.3. Distribution of cementite

Apart from the processes of spheroidization and coarsening of cementite, which are typical for eutectoid steels [1], the process of a homogeneous distribution of cementite after the spheroidization in the present ferritic–pearlitic steel have been observed. This means that after HWD with a subsequent coiling even within the former proeutectoid ferrite regions cementite particles can also be found. The various stages of the cementite redistribution after a cooling rate of 10 K/s and deformation/coiling temperatures 873-943 K (600–670 °C) are shown in Fig. 3. At an early stage of the redistribution process, accomplished here by using a rather low

deformation/coiling temperature of 873 K (600 °C) (cf. Fig. 3(a)), the microstructure contains proeutectoid ferrite without cementite particles and fine spheroidized cementite particles (with a size of about 0.1 µm) that are located at the areas of former pearlite colonies. So, after a complete spheroidization the distribution of cementite is not homogeneous. However, during the deformation/ coiling at higher temperatures, the fine cementite particles may dissolve and some carbon atoms are assumed to diffuse from the areas of the former pearlite colonies to the cementite free areas of the former proeutectoid ferrite followed by a subsequent reprecipitation and coarsening (cf. Fig. 3(b) and (c)). As a result (cf. Fig. 3(d)) the cementite particles with a size of about 1 µm are distributed rather homogeneously in the ferritic matrix.

As reported in [9,10], the phenomenon of pearlitecolony dissolution during the annealing of a low carbon titanium microalloyed steel after cold rolling [9] and low carbon vanadium microalloyed steel after severe plastic deformation [10] was observed. In these cases the microalloying elements cause an increase in the recrystallization temperature. The main condition for the redistribution of cementite, as shown in [10], seems to be a high dislocation density in the heavily deformed former pearlite colonies that was estimated to be of the order of 10^{16} m⁻². In case of plain low carbon steel [9,10] with low recrystallization temperature, these



Fig. 3. Effect of deformation/coiling temperature on cementite distribution after cooling rate 10 K/s: (a) 873 K (600 °C); (b) 923 K (650 °C) and (c,d) 943 K (670 °C).

treatments did not lead to the disappearance of pearlite colonies. In this steel a recrystallization would slow down or even stop this process by significantly reducing the dislocation density. This means that a higher recrystallization temperature offers the possibility that even at fairly high temperatures, when the cementite dissolution already starts, only recovery takes place. In this situation the dislocation density will not be reduced very much, so that the redistribution of cementite can take place rapidly assisted by a fast dislocation pipe diffusion. On the other hand, in the case of a low recrystallization temperature the recrystallization begins before the start of cementite dissolution, the dislocation density decreases drastically and a decomposition of colonies does not take place.

A driving force for the redistribution can be a gradient of the solute carbon. Inside the former colony around the fine cementite particles the carbon concentration is essentially higher compared to the proeutectoid ferrite. The high density of dislocations and vacancies during or after HWD facilitates the solute carbon diffusion to areas of lower carbon concentration, i.e., proeutectoid ferrite, with subsequent reprecipitation in the most energetically favorable places such as triple joints of ferrite grain boundaries (cf. Fig. 3(d)). Additionally, due to the faster grain boundary diffusion, the particles located on grain boundaries and triple junctions will have a size advantage in the later Ostwaldripening process.

3.4. Softening of ferrite

The various stages of ferrite softening after a cooling rate of 10 K/s and deformation/coiling temperatures in the range of 903-983 K (630-710 °C) are shown in Fig. 4(a). After deformation at 903 K (630 °C) and coiling at the same temperature, the formation of numerous subgrains with low angle boundaries can be observed. These subgrains remain inside the original deformed grains without growth into neighboring grains. Considering only the high angle grain boundaries, it is obvious that the grains are highly elongated in the rolling direction. The amount of high angle grain boundaries (with misorientation angle $>15^{\circ}$) is about 50%. After the increase of the deformation/coiling temperature up to 983 K (710 °C), the fraction of high angle grain boundaries increases up to 65–70%, and the microstructure contains fine equiaxed ferrite grains. However, it seems that most of these grains keep the original orientations of the former deformed grains. For example, the areas with the orientation near $\{111\}$ (cf. Fig. 4(a), single arrow) or near $\{001\}$ (cf. Fig. 4(a), double arrow) consist of fine subgrains with low angle boundaries, which are characteristic for the microstructure up to the highest studied subcritical temperature 983 K (710 °C).

The ODF sections in Fig. 4(b) show that the texture of the steel essentially does not change after the various deformation/coiling temperatures, containing mainly γ



Fig. 4. EBSD images (a) and ODF section $\phi_2 = 45^\circ$ (b) after the various deformation/coiling temperatures.

and α -fibers with a maximum near $\{1\,1\,2\}\langle 1\,1\,0\rangle$, which is typical for rolling texture [11,12].

The distribution of grain/subgrain boundaries misorientations (Fig. 5) for the deformation/coiling temperatures 903 K (630 °C) and 983 K (710 °C) are very similar. They both show a high fraction of low angle grain boundaries with small misorientation angles. The increase of the temperature by 80 K leads only to a continual decrease of the low angle fraction (<15°) with a simultaneous increase of the fraction of high angle boundaries. (Because of the orientation noise in the EBSD measurements misorientations less than 1.5° have been omitted).

The distribution of the ferrite grain size (Fig. 6) shows also a continuous change of the microstructure with increasing HWD temperature. After the temperature $630 \ ^{\circ}C \ (903 \ K)$, the absolute maximum of the area fraction corresponds to the grain size of around 2 µm. A



Fig. 5. Distribution of grain/subgrain boundaries misorientations.



Fig. 6. Area fraction of ferrite grain size after the various deformation / coiling temperatures.

further local maximum near 6 μ m can be attributed to the large deformed ferrite grains still without substructure. After the deformation/coiling at 923 K, the curve exhibits a fairly sharp maximum for grain sizes around 2.2 μ m. The further increase of the temperature leads to a shift of the maximum area fraction to larger grain sizes up to 3.5 μ m for 983 K.

Thus, the results of the study suggest that the ferrite softening during the deformation with the subsequent coiling can be attributed to recovery processes (i.e., polygonization), called also continuous recrystallization or recrystallization in situ. In this case the subgrains form within the deformed matrix and grow, so that the dislocation density decreases due to the reduction of subgrain boundaries area and, finally, formation of high angle grain boundaries.

The reasons that only recovery and not primary recrystallization takes place here may be described as follows. Due to the lamellae spheroidization, a fine dispersion of cementite particles are present in the microstructure. These particles lead to a high dragging force for the migration of high angle grain boundaries due to Zener pinning of the boundaries that increases the recrystallization temperature. On the other hand, it is known that due to high stacking fault energy the recovery in ferrite can proceed very quickly. In this case the dislocation-rearrangement to form energetically more favorable configurations starts everywhere, but the subsequent migration of high angle grain boundaries may be stopped very soon by the particles.

The recovery process decreases both the stored energy and the local stored energy gradient, which slows down the successful nucleation or growth. Moreover, an increased amount of solute carbon in ferrite during the HWD and the redistribution of cementite due to the spheroidization process can retard both the formation and migration of high angle boundaries.

At higher temperatures, the homogeneously distributed relatively fine cementite particles produce a stabilizing effect on the fine grained ferrite matrix. But the coarsening of the cementite particles leads to a reduction of the Zener drag effect, so that primary recrystallization and grain growth can occur.

Because the recovery processes involve a short-range interaction between dislocations and subgrain boundaries, or between adjacent boundaries, they may lead to a sharpening of deformation texture and a higher intensity of deformation texture components, which can be seen in Fig. 4(b).

The start of the formation of low angle subgrain boundaries due to dislocation rearrangement in a deformed grain is shown in Fig. 7(a) (EBSD image and misorientation profile). The initial deformed grain with the main orientation component near $\{111\}$, with a length of more than 10 µm and a width in normal direction of 1-1.5 µm contained initially (after the deformation) a high density of excess dislocations, which resulted in a fairly high long-distance misorientation gradient $\sim 0.5-1^{\circ}/\mu m$ along the rolling direction. The climb and cross slip of dislocations lead to their rearrangement as low angle boundaries. The misorientation profiles allow observation the beginning of subgrain formation. The plateaus at the "point-to-origin" profile evidently can be attributed to new subgrains with low angle boundaries near 1°, as shown in the "pointto-point" profile.

The formation of low angle subboundaries in progress is shown in Fig. 7(b). In the initial deformed grain with an orientation component near $\{1\,1\,2\}$, the low angle boundaries between new subgrains already have higher misorientation angles up to 9°, with a subgrain size of about 1–3 µm.

An increase in temperature leads to the continuous decrease of the fraction of low angle boundaries along with a simultaneous increase of high angle ones (cf. Fig. 5). As a result, the amount of high angle boundaries



Fig. 7. Formation of (sub)grain boundaries during continuous recrystallization: (a) start of low angle subgrain boundaries formation; (b) formation of low angle subgrain boundaries in progress and (c) formation of high angle grain boundaries due to dislocation accumulation on low angle boundaries.

increases up to \sim 70%. As shown in literature, the continuous recrystallization clearly contributes to the formation of new high angle boundaries. The possible mechanisms are the accumulation of dislocations at the subgrain boundaries [13], the increase of misorientation angle by the merging of lower angle boundaries during subgrain coalescence [14,15] and the subgrain growth with migration of low angle boundaries via dislocation motion [16]. In the postmortem analysis carried out in this work it is not possible to prove exactly, which mechanism is really active or relevant for the microstructure development. Nevertheless, some of the interesting features found in the microstructures will be discussed to illustrate the possible mechanisms of continuous recrystallization in studied steel.

The increase of boundary misorientation that can be attributed to the accumulation of dislocations into subboundaries is shown in Fig. 7(c). A strained grain is separated into several subgrains of similar orientation around $\{1\,1\,1\}$. One of these (sub)grains already exhibits misorientations of up to 25° to the neighboring (sub)grain.

Subgrain coalescence is often reported to be a very slow process. Nevertheless, the microstructure in Fig. 8(a) might be contributed by the coalescence of two subgrains with orientation near {111} and misorientation $\leq 1^{\circ}$ (see misorientation profile, Fig. 8(b)). As reported in [17], the coalescence can be favorable in case of joining subgrains surrounded by high angle boundaries. This condition is completely fulfilled for subgrains A1 and A2, which are surrounded by high angle boundaries ranging from 20° up to 47°. Preferential subgrain coalescence may occur since the driving force for subgrain merging comes from the difference between the higher energy of dislocations in the low angle boundary and the lower energy of the dislocations in the high angle boundaries. Since the high angle boundaries around two subgrains with low misorientation do not occur often, the coalescence of subgrains is observed



Fig. 8. The processes of two subgrains coalescence: (a) EBSD image, (b) misorientation profile and (c) subgrain growth (EBSD image); the misorientation angles between grains are shown.

only rarely, compared to other mechanisms of continuous recrystallization.

The possible subgrain growth is shown in Fig. 8(c). The curvature of the boundaries indicates the growth of the subgrain A with orientation near $\{1\,1\,1\}$ at the expense of the adjacent (sub)grains (see arrows). Apart from the high angle boundaries migration (between grain A and the each of grains B, D and F, the boundaries have misorientations of about 20°), the migration of low angle boundaries can be observed (AC 12°, AE near 9° and the boundary between A and G has a misorientation angle only about 3°).

As reported in [18], low angle boundaries in general have a low mobility. The reason is the different structure of high and low angle boundaries. For high angle boundaries, migration can occur easily by single atom jumps across the rather open grain boundary structure. Low angle boundaries composed of dislocation arrays migrate by the motion of dislocations, i.e., a coordinated movement of atoms. While the motion of an individual dislocation by glide is easy, the motion of an array might be more difficult, since motion of the arrays causes a change in shape of the two subgrains, which is resisted by surrounding subgrains. As reported in [18], the limitative process of the array motion is the climb of edge dislocations and the activation energy of the process has the order of magnitude of the activation energy for self-diffusion. On the other hand, as shown in [19], even a rather low deformation leads to a decrease of the self-diffusion activation energy.

The activation energy of the (sub)grain growth with the increase of the deformation/coiling temperature was estimated using the (sub)grain size with the maximum area fraction (cf. Fig. 6).

The estimation was carried out based on the following considerations. As shown in [18], for isothermal annealing a time law of the form

$$d = (at)^n \tag{1}$$

is frequently found, where d is grain size, t is time, and n and a are constants. As reported in [20,21], $n \le 0.5$, in [19] n = 0.5.

As shown in [19,21] the increase in temperature leads to the exponential increase of constant a that has the nature of a diffusion coefficient [19]

$$a = a_0 \exp(-Q/RT),\tag{2}$$

where Q is the activation energy of grain growth, T is the absolute temperature and a_0 is constant. For isochronous conditions

$$d = a_0^n \exp(-nQ/RT),\tag{3}$$

and a plot of $\ln d$ versus 1/T allows for determining the activation energy Q of grain growth from the gradient (-nQ/RT).

The plot of $\ln d$ versus 1/T is shown in Fig. 9. It can be seen that for temperatures 903–963 K (630–690 °C), the process has a lower activation energy compared to the grain growth in a range 963–983 K (690–710 °C). Here, it should be mentioned that the point for 903 K (630 °C), where the process of subgrain formation only starts, apparently can be assigned to the line 923-963 K (650-690 °C), where the process is in progress. Assuming that n = 0.5, the activation energies at lower and higher temperature ranges can be calculated as 71 and 205 kJ/ mol, respectively. According to Kristal [19], the lower Qcan be attributed to the activation energy of interstitial elements diffusion (carbon or nitrogen), the higher Q has the order of magnitude of the self-diffusion activation energy of deformed ferrite. This might mean that the limiting factor of ferrite softening at the temperatures up to 963 K (690 °C) is carbon diffusion during the spheroidization and distribution of cementite.

3.5. Effect of cementite on ferrite softening

690

Q~205 J/mol

0.00105

710

1.4

1.3

1.2

1.1

1

0.9 0.8

0.7

0.6

0.001

ln d, µm

Just after the spheroidization (cf. Fig. 3) the size of cementite particles can be about 0.1 µm. The presence of these fine cementite particles retards the softening of ferrite in the areas of the former pearlite colonies. The microstructure of steel with fine cementite is shown in

650

630

Q~71 J/mol

0.0011

T, °C

0.00115

Fig. 9. Different stages of ferrite grain growth.

1/T, K⁻¹

Fig. 10. Effect of cementite on ferrite softening process: (a) SEM and (b) EBSD image.

SEM and EBSD images of the same location in Fig. 10. It can be seen that in the area of fine cementite the formation of subgrains is not complete (cf. Fig. 10, area A). On the contrary, in the area of proeutectoid ferrite, the rearrangement of dislocations with the formation of low angle subgrain boundaries is in progress (cf. Fig. 10, area B).

This confirms that the limitation factor of ferrite softening can be the diffusion of carbon atoms. As shown in [15], for two-phase alloys the decomposition before recrystallization is controlled by the lower activation energy for diffusion of interstitial atoms (carbon) as compared to that which controls primary recrystallization (self-diffusion of iron atoms). The processes of spheroidization and distribution of cementite might be assumed to be decomposition, since they can be attributed to a local change of chemical composition, i.e., the change of carbon content in solid solution during lamellae dissolution, cementite re-precipitation, as well as dissolution of the fine particles with a coarsening of larger ones. Obviously, the formation and growth of ferrite subgrains are controlled by solute carbon and cementite particles, which grow by diffusion of carbon. The particle spacing determines the size of subgrains.

3.6. Effect of initial microstructure on cementite distribution and ferrite softening

As shown in Fig. 1, various cooling rates allow for obtaining various initial microstructures before the HWD. The effect of the cooling rate, i.e., the initial microstructure on the microstructure after HWD was studied.



The microstructures after HWD of steels with two different cooling rates and deformation/coiling temperatures 10 K/s, 943 K (670 °C) and 20 K/s, 973 K (700 °C) are shown in Fig. 11. In the case of low cooling rate (10 K/s), the microstructure after transformation is composed of proeutectoid ferrite and pearlite. With this initial microstructure, the HWD and subsequent simulated coiling (cf. Fig. 11(a)) results in a homogeneous distribution of cementite particles in a fine grained ferrite matrix, according to processes, shown and discussed earlier. According to the EBSD measurement, the ferrite contains about 65% high angle grain boundaries indicating advanced progress of continuous recrystallization. The increase of the cooling rate up to 20 K/s leads to a microstructure with proeutectoid ferrite, a finer pearlite and bainite, which results in areas with accentual finer cementite particles after HWD even at higher deformation/coiling temperature of 973 K (700 °C). These very fine cementite particles retard a subgrain formation in these areas (cf. Fig. 10). As a result the final microstructure is inhomogeneous and contains only 45% high angle ferrite grain boundaries (cf. Fig. 11(b)).

3.7. Coarsening of ferrite

The microstructure with fine ferrite (sub)grains that is stabilized due to homogeneously distributed cementite particles is fairly stable even after the deformation/ coiling at high temperatures of ferritic range. But by some unfavorable circumstances some enlarged subgrains can abnormally grow.

Such a situation can occur in an inhomogeneous initial microstructure after a higher cooling rate near 20 K/s (cf. Fig. 11(b)). In this case, a local stored energy gradient may facilitate the preferential growth of one or several grains with high angle boundaries, as shown in Fig. 12. The grains in the areas of the former proeutectoid ferrite (cf. Fig. 10, area B), with coarser cementite particles, can be such potential fast growing grains. This abnormal grain growth is enhanced if the growing grain is surrounded by areas with essential higher stored energy, i.e., subgrains with low angle boundaries and partly deformed grains. The energy lowering due to the consumption of these areas by the growing coarse grain provides the driving force for this process.



Fig. 11. Microstructure after the various cooling rates and deformation/coiling temperatures: (a) 10 K/s, 943 K (670 °C) and (b) 20 K/s, 973 K (700 °C).



Fig. 12. Coarsening of ferrite: (a) SEM and (b) EBSD image.

4. Conclusions

- Spheroidization of pearlite during the HWD is accelerated by the formation of cementite lamellae kinks, fracture of lamellae and cementite subboundaries. Higher equilibrium carbon concentration in ferrite (higher carbon solubility) near these lamellae defects provokes a quick local dissolution of the lamella that leads to its division with a subsequent or simultaneous spheroidization.
- 2. A rather homogeneous distribution of cementite in an initially ferritic-pearlitic microstructure can be observed due to increase of deformation/coiling temperatures to 943–973 K (670–700 °C). This process is assumed to proceed by the local partial dissolution of former pearlite colonies due to a local increase of carbon solubility in ferrite. Simultaneously, the solute carbon may diffuse into the cementite-free proeutectoid ferrite areas with lower equilibrium carbon concentration and re-precipitate. Both of these processes are supported by a high density of lattice defects due to HWD.
- 3. Ferrite softening during the HWD can be attributed to a continuous or in situ recrystallization. Primary recrystallization is hardly probable because of the relatively low stored energy at rather high deformation temperatures as well as an increased amount of solute carbon in ferrite during the cementite spheroidization and fine cementite particles produced by spheroidization.
- 4. Continuous recrystallization contributes to high angle boundaries development due to accumulation of dislocations at the subgrain boundaries, by the subgrain growth with migration of low angle boundaries and by the merging of lower angle boundaries during subgrain coalescence.
- 5. According to the determination of activation energy of (sub)grain growth, the controlling factor of ferrite softening in the temperature range 903–963 K (630–

690 °C) is carbon diffusion during the spheroidization and redistribution of cementite.

- 6. The HWD of steel with initial ferrite–pearlite microstructure (after cooling rate 10 K/s) results in a homogeneous distribution of cementite in a fine grained ferrite matrix with about 65–70% high angle grain boundaries. On the contrary, the HWD of steel with initial ferrite– pearlite–bainite microstructure (after cooling rate 20 K/s) brings about an inhomogeneous cementite distribution with the areas of fine cementite particles and only 45% high angle ferrite grain boundaries.
- 7. In case of inhomogeneous microstructure (cooling rate 20 K/s), a high local gradient in the size of cementite particles tends to facilitate the preferential growth of some grains with mobile high angle boundaries, which leads to microstructure coarsening.

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References

- [1] Kaspar R, Kapellner W, Lang C. Steel Res 1988;59(11):492.
- [2] Robbins J, Shepard O, Sherby O. J Iron Steel Inst 1964;202(Oct.):804.
- [3] Pawelski O, Kaspar R. Materialprüfung 1988;30(11-12):357.
- [4] Chattopadhyay S, Sellars S. Acta Met 1982;30(1):157.
- [5] Watanabe T, Karashima S. Physica Status Solidi 1970;42:749.
- [6] Chalmers B. Physical metallurgy. New York: Wiley; 1959.
- [7] Novikov I. Theory of heat treatment of metals. Moscow: Metallurgia; 1986. p. 479.
- [8] Paqueton H, Pineau A. JISI 1971;209(12):991.
- [9] Babich V, Nesterenko A, Storojeva L. Met Sci Heat Treat (USSR) 1989;31(9–10):692.
- [10] Shin D, Park K, Kim Y. Scripta Mater 2003;48(5):469.

- [11] Hutchinson B, Bate P. In: Proceedings of the International Conference on Properties and Application of IF Steels. Tokyo: ISIJ; 2003. p. 337.
- [12] Quadir M, Duggan B. In: Proceedings of the International Conference on Properties and Application of IF Steels. Tokyo: ISIJ; 2003. p. 357.
- [13] Hales S, McNelley T, McQueen H. Metall Mater Trans A 1991;22A:1037.
- [14] Hornbogen E, Köster U. In: Haessner F, editor. Recrystallization of metallic materials. Berlin: Dr. Riederer Verlag; 1978. p. 159.
- [15] Hornbogen E. Metall Mater Trans A 1979;10A:947.
- [16] Nes E. In: Baudelet B, Surey M, editors. Superplasticité. Paris: Centre National de la Recherche Scientifique; 1985. p. 7.1.
- [17] Doherty R, Cahn R. J Less-Common Metals 1972;28:279.
- [18] Haessner F, editor. Recrystallization of metallic materials. Stuttgart: Dr. Riederer Verlag; 1978. p. 293.
- [19] Kristal M. Diffusion processes in iron alloys. Moscow; 1963. p. 278.
- [20] Holmes E, Winegard W. Acta Met 1959;7:411.
- [21] Drolet J, Galibois A. Acta Met 1968;16:1387.





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Review

Overview of processing, microstructure and mechanical properties of ultrafine grained bcc steels

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Abstract

Ultrafine grained steels with grain sizes below about 1 μ m offer the prospect of high strength and high toughness with traditional steel compositions. These materials are currently the subject of extensive research efforts worldwide. Ultrafine grained steels can be produced either by advanced thermomechanical processes or by severe plastic deformation strategies. Both approaches are suited to produce submicron grain structures with attractive mechanical properties. This overview describes the various techniques to fabricate ultrafine grained bcc steels, the corresponding microstructures, and the resulting spectrum of mechanical properties.

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Keywords: Ultrafine grained steels; Microstructure; Thermomechanical processing; Severe plastic deformation; Mechanical properties

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1. Introduction

Among the different strengthening mechanisms, grain refinement is the only method to improve both strength and toughness simultaneously. Therefore, ultrafine grained steels with relatively simple chemical compositions, strengthened primarily by grain refinement, have great potential for replacing some conventional low alloyed high strength steels. The main benefits behind such an approach are to avoid additional alloying elements; to avoid additional heat treatments like soft annealing, quenching and tempering; and to improve weldability owing to lower required carbon contents and other alloying elements when compared with other high strength steels. A further high potential domain for such ultrafine grained steel is the possibility for high strain rate superplasticity at medium and elevated temperatures [1]. In general, the term ultrafine grain is used here in the context of average grain sizes between 1 and 2 µm in diameter; submicron refers to grain sizes between 100 and 1000 nm; while nanostructured refers to grain sizes below about 100 nm.

The purpose of this overview is to provide a detailed introduction to the processing technologies, to the resulting microstructures, and to the mechanical properties associated with ultrafine grained body centered cubic (bcc) steels.

2. Methods of producing ultrafine grained steels

2.1. Introduction

Currently, laboratory techniques to produce ultrafine grained bcc steels utilize two approaches: severe plastic deformation techniques or advanced thermomechanical processing, which essentially involves modification to conventional large scale steel rolling processes. Compared to severe plastic deformation techniques, advanced thermomechanical methods are largescale industrial processes and can be somewhat more readily optimized to operate in temperature regimes where they beneficially exploit phase transformation and controlled cooling.

2.2. Severe plastic deformation

2.2.1. Severe plastic deformation techniques for steels

Severe plastic deformation (SPD) techniques [2–4] impose large accumulated plastic strains at room or elevated temperatures, e.g. mainly in the temperature regime of warm deformation. These techniques can be used to produce ultrafine grained steels with an average grain size below 1 μ m [5–19]. Typical SPD techniques include equal-channel angular pressing (ECAP) [5–11], accumulative roll bonding (ARB) [12–14], bidirectional compression [15], and high-pressure torsion (HPT) [16–19].

2.2.2. Equal-channel angular pressing

Equal-channel angular pressing imposes large plastic strains on massive billets via a pure shear strain state. The approach was developed by Segal et al. in the early 1980s [20]. Its goal was to introduce intense plastic strain into materials without changing the cross-sectional area of the deformed billets. Owing to this characteristic, repeated deformation is possible. At the beginning of the 1990s this method was further developed and applied as a severe plastic deformation method for the processing of microstructures with submicron grain sizes [21]. The equalchannel angular pressing method was mainly applied for nonferrous alloys (e.g. Al and Mg alloys) and some low carbon steels. The finest ferrite grain size obtained by use of this method is reportedly about 0.2 μ m [7,22].

2.2.3. Accumulative roll bonding

Accumulative roll bonding essentially involves repeated application of conventional rolling. This approach has been suggested to possess the potential for mass production [12–14,23–25]. While rolling is an attractive deformation process for continuous production of bulk sheets, the total reduction in thickness, i.e. the accumulated strain, which can be achieved by this method, is limited because of the decrease in the strip thickness with increasing rolling reduction. In order to obtain bulk material, rolled sheets are stacked and then bonded together during rolling. Hence, the process involves simultaneous bonding and deformation. In the accumulative roll bonding method, the rolled material is cut, stacked to the initial thickness and rolled again. Owing to this approach, multiple repetitions are possible to achieve huge strains. A natural limit of this approach lies in the increase in strength and the gradually reduced surface quality of the roll-bonded sheets.

2.2.4. High pressure torsion

High-pressure torsion (HPT) imposes a pressure of up to several GPa for the fabrication of disk shaped samples with a diameter from 10 to 20 mm and a thickness of 0.2–0.5 mm [19]. A disk shaped specimen, which is usually first provided as a powder sample, is compressed in an almost closed die. During loading, the contact platens rotate in opposite directions in order to impose a shear strain. The through-thickness distribution of shear strain depends on the contact friction, a function of the roughness of the contact plates and the lubrication state. The torsion straining achieves a substantial degree of substructure refinement and controls the evolution of large crystallographic misorientations among adjacent grains. The HPT technique also has the advantage of being able to refine the grain size during powder consolidation, making it possible to produce bulk nanomaterials from micrometer-sized metallic powders.

2.2.5. Bi-directional large strain deformation

Bi-directional compression can be used to introduce large plastic strains in steels. It combines severe plastic deformation (large strain) and thermomechanical processing (phase transformation and controlled cooling can be exploited). Compression is realized by alternate forging in two perpendicular directions. Elongation in the third direction is usually not restricted.

2.3. Advanced thermomechanical processes

2.3.1. Introduction

In contrast to severe plastic deformation approaches in which large strain is the main factor, advanced thermomechanical processes pursue alternative strategies to produce ultrafine ferrite grains. For instance, these processes exploit dynamic recrystallization of austenite during hot deformation with subsequent $\gamma \rightarrow \alpha$ (austenite to ferrite) transformation [26]; strain-induced ferrite transformation (i.e. transformation during rather than after deformation) [27–32]; hot rolling in the intercritical region (i.e. in the austenite/ferrite two-phase region) [33]; warm rolling in the ferrite region [34] involving either dynamic recrystallization or pronounced recovery of the ferrite during warm deformation [35–45]; or cold rolling and annealing of a martensitic starting microstructure [46–51].

2.3.2. Recrystallization of austenite during hot deformation

An important mechanism that is widely used for grain refinement in steels is dynamic recrystallization during hot deformation [26]. This technique has been used to produce ferrite grain sizes as fine as 2-5 µm via recrystallization-controlled rolling or by conventional rolling followed by accelerated cooling. In recrystallization-controlled rolling fine precipitates restrict austenite grain growth after deformation. Recrystallizationcontrolled rolling is often used in conjunction with accelerated cooling and microalloying in order to effectively refine the grain size. Accelerated cooling is used to increase the cooling rate through the transformation zone in order to decrease the transformation temperature. In principle, a lower transformation temperature results in a higher ferrite nucleation rate due to a higher undercooling, and a decreased growth rate. Conventional controlled rolling has been implemented in many commercial operations through the addition of elements such as Nb, which increases the recrystallization temperature to over 1173 K, such that deformation in the last passes are applied below the recrystallization temperature. This increases the density of sites for ferrite nucleation.

2.3.3. Strain-induced ferrite transformation

A simple rolling procedure which entails strain-induced phase transformation from austenite to ferrite has been found to provide significant grain refinement in the sheet surface. In this approach, steel strips are reheated to obtain austenite microstructure and subsequently rolled in a single pass (30% reduction) just above A_{r3} (austenite to ferrite transformation temperature) but below A_{e3} (equilibrium austenite to ferrite transformation temperature) [27,52–56]. The three critical factors promoting the formation of ultrafine ferrite grains during a strain-induced transformation are a high shear strain, a high cooling rate as a result of rapid heat transfer to the colder rolls during the roll pass, and an appropriate deformation temperature (between A_{r3} and A_{e3}).

Hodgson et al. [27] applied strain-induced transformation to a plain carbon steel strip (0.06C–0.59Mn, wt.%) with an original thickness of about 2 mm, reduced to about 1.4 mm after a single pass at roll exit temperatures between 953 and 983 K. Equiaxed ultrafine ferrite grains of about 1 μ m in the subsurface region were obtained, but the microstructure of the rolled strip was inhomogeneous through the thickness. The microstructure consisted of ultrafine ferrite grains in the surface layers, which penetrated to between one-quarter and one-third of the thickness with coarser ferrite (about 5–10 μ m) and pearlite in the core of the strip.

2.3.4. Intercritical hot rolling

Ultrafine ferrite grains in plain C–Mn steels have also been obtained through hot rolling in the intercritical region (i.e. in the austenite plus ferrite two-phase region) by Yada et al. [57]. They attributed grain refinement to both dynamic transformation of austenite into ferrite and the dynamic recrystallization of the ferrite phase. Nucleation of ferrite at austenite grain boundaries during the dynamic transformation was considered to play a major role in the formation of ultrafine ferrite grains while dynamic recrystallization of ferrite was assumed to be of minor relevance.

2.3.5. Dynamic recrystallization of ferrite during warm deformation

Warm deformation in the ferrite regime may further refine steel microstructures that were previously refined during transformation. It has been considered that recovery is the main softening process during warm deformation of ferrite and that dynamic recrystallization does not occur [35]. This behavior is attributed to the fact that bcc ferrite has a high stacking fault energy which results in rapid recovery and insufficient accumulation of stored deformation energy to promote dynamic recrystallization. However, the occurrence of dynamic recrystallization of ferrite has been reported by several researchers [35–37,58]. The recent study of Murty et al. [36] confirmed the occurrence of dynamic recrystallization of ferrite in an ultralow carbon steel processed by warm deformation at a strain rate of 0.01 s⁻¹ (low Zener–Hollomon parameter). Since warm deformed ferrite usually contains pronounced subgrain structures that are sometimes difficult to distinguish from recrystallized grains in standard light optical micrographs, the authors confirmed the occurrence of dynamic recrystallization in ferrite by use of the electron backscattered diffraction (EBSD) technique to characterize the crystallographic relationships across grain boundaries. Most of the equiaxed ferrite grains were surrounded by high-angle grain boundaries (HAGBs) (with grain boundary misorientations $\geq 15^{\circ}$) rather than by lowangle grain boundaries (with grain boundary misorientations <15°).

In another case, warm-rolling of interstitial free (IF) steel in the ferrite region was found by Najafi-Zadeh et al. [34] to produce ultrafine ferrite with grain size of $1.3 \,\mu\text{m}$. Dynamic recrystallization of ferrite was considered to play a major role in the formation of ultrafine ferrite. A key barrier to the occurrence of dynamic recrystallization of ferrite is suggested to involve the presence of interstitial elements such as C and N. Removing interstitial elements from the matrix reduces the possibility of strain-induced precipitation, which inhibits dynamic recrystallization and increases the likelihood of dynamic recrystallization of ferrite [34].

2.3.6. Pronounced recovery of ferrite during warm deformation and annealing

Recently, Song et al. [39–45] have reported the production of ultrafine ferrite through pronounced recovery following warm deformation and annealing. Compared with the earlier studies on low carbon ultrafine grained bcc steels, Song et al. [39-45] investigated medium carbon steels in an effort to increase the work hardening rate of ultrafine grained steels, since high work hardening rates are associated with high ductility. In their studies, steels with ultrafine ferrite grains and homogeneously distributed cementite particles were produced by large strain warm deformation ($\varepsilon = 1.6$) at 823 K and subsequent annealing (Fig. 1). The ultrafine microstructures obtained were stable against grain and cementite coarsening even during a 2h annealing treatment at 823 K. Pronounced recovery instead of primary recrystallization was required to obtain a large fraction of HAGBs. It was concluded in [39] that the prevalence of primary recrystallization, instead of recovery, is not generally beneficial in warm rolling. Primary recrystallization reduces significantly the dislocation density and removes the substructure, which is important for the gradual formation of subgrains that eventually become ultrafine grains surrounded by HAGBs.

2.3.7. Cold rolling and annealing of martensitic steel

Another route to fabricate ultrafine grained steel was developed by Tsuji et al. [49–51]. The process includes cold-rolling (50% reduction) of a martensite starting microstructure in a low carbon steel (0.13 wt.% C) and subsequent annealing at 773–873 K. The final microstructure was reported to consist of ultrafine ferrite grains and uniformly precipitated carbides. The formation of an ultrafine microstructure was attributed to the fine martensite starting microstructure, which augmented the effect of plastic deformation enhancing grain subdivision [49–51]. The high dislocation density as a result of cold rolling and the high concentration of solute carbon atoms in the martensite were also expected to facilitate grain subdivision by causing inhomogeneous deformation [49–51].

2.4. Summary of the two strategies of producing ultrafine grained steels

2.4.1. Differences

As mentioned above, ultrafine grained steels can be produced by two main methods. Table 1 gives a summary of the various process techniques described above and the ferrite grain sizes obtained for the different bcc steels. Among the SPD techniques the accumulated plastic strains (true strains) required to obtain submicron-sized grains are of the order of 3–4 using ECAP and of the order of 5–6 using the ARB process. For the SPD methods, a well-designed strain path is more important and also more feasible than a precisely controlled temperature path. The small-scale complexity and the "batch" nature of these methods suggest that they would require considerable ingenuity and investment for application to high volume steel production.

The advanced thermomechanical processing routes employ a relatively low accumulated strain in the range of about 1.0–3.6 to produce ultrafine grained steels (except for the strain-induced ferrite transformation technique which typically requires even less strain). The advanced thermomechanical processing methods are less effective with respect to grain refinement, but more adaptable to large sample sizes when compared with the



Fig. 1. SEM image (a) and EBSD map (b) after large-strain deformation ($\varepsilon = 1.6$) and subsequent 2 h annealing at 823 K obtained for a plain C–Mn steel (CD, compression direction; TD, transition direction). The black lines indicate grain boundary misorientations between 15° and 63°. White lines indicate grain boundary misorientations between 2° and 15°. (c) TEM micrograph of an ultrafine grained steels after large strain warm deformation ($\varepsilon = 1.6$, and 2 h at 823 K) with 0.74 mass% Mn. Arrows "1" point at very fine cementite particles inside the ferrite grains; arrows "2" point at coarse cementite particles at the ferrite grain boundaries. (d) Corrresponding TEM micrograph for a steel with 1.52 mass% Mn. Details of the compositions and of the processing are given in [39–45].

SPD methods. An important issue in this context, however, is that in the case of large sample sizes the strain and cooling paths have to be carefully controlled since they are key parameters that govern the final grain size within relatively small process windows.

A further difference between these two approaches is that the advanced thermomechanical methods are continuous processes and can be well optimized when they work in a temperature regime where they exploit phase transformation and controlled cooling. The most significant feature of transformation refinement is the possibility of optimizing the conditions to produce a maximum number of new grains that usually nucleate at grain boundaries. In this context, the high temperature phase can be pretreated to increase the grain boundary area (refined or pancaked grains) and to introduce a dense dislocation substructure by large strains at the lowest possible temperature to avoid static primary recrystallization. Ultimately, the transformed product can be subjected to warm or cold deformation, possibly in conjunction with precipitation of carbides in steel. A concern in this context is not only the desired increase in strength but also the possible drop in toughness and ductility [61].

2.4.2. Similarities

Ultrafine grained ferrite microstructures are of great interest for low alloyed structural steels as reflected by the steels reported in Table 1, regardless of severe plastic deformation or advanced thermomechanical processes. Structural steels with improved mechanical properties may facilitate light-weight construction design (buildings, bridges, large structures). Both the SPD and advanced thermomechanical processes may encounter difficulties in being scaled up to large commercial scales and

Table 1 Summary of different techniques reported to produce ultrafine grains in bcc steels

Techniques	Steels	Steels composition (wt.%)	Ferrite grain size achieved (µm)	Log. strain imposed [1]	Deformation temperature (K)	Heat treatment after deformation	Reference
	Plain low carbon steel	0.08C-0.42Mn-0.18Si	0.2	3.0	293	AC	[7]
ECAD	Plain low carbon steel	0.15C-1.1Mn-0.25Si	0.3 in thickness	4.0	623	AC	[22]
ECAP	Ti-V carbon steel	0.1C-1.59Mn-0.29Si-0.02Ti-0.05V	~ 0.3 in thickness	1.0	573	AC	[59]
	Ferrite-martensite dual phase steel	0.15C-1.06 Mn-0.25Si	0.8	4.0	773	$1003~\text{K} \times 10~\text{min}~\text{WQ}$	[46]
ARB	Ti added IF steel	0.003C-0.15Mn-<0.01Si-0.049Ti	0.4	5.6	773	WC	[14]
НРТ	Plain low carbon steel	~0.7C-~1.0Mn-~0.3Si	0.01	Shear strain 300, log. strain 0.45	293	AC	[16,18]
DRX [*] during hot deformation	Microalloyed steel	0.11C-1.45Mn-0.34Si-0.068Nb	2–5	Final rolling 2.2–3.6	1153–1033	AC	[26]
Strain-induced ferrite transformation	Plain low carbon steel	0.06C-0.59Mn	1.0 (strip surface)	0.36	1053	AC	[27]
Deformation in the intercritical region	Plain low carbon steel	0.17C-1.32Mn-0.44Si-0.15Nb	2.1	2.3	973	WQ	[60]
Warm rolling in the ferrite region	Ti added IF steel	0.003C-0.15Mn-0.022Si-0.065Ti	1–3	Final rolling $\sim 0.55 \times 5$	Below A_{r1}^*	WQ	[34]
DRX [*] of ferrite during warm deformation	Ultra-low carbon steel	0.0016C-0.1Si-0.3Mn	-	4.0	723–823 (lower than A_{c1}^{*})	WQ	[36]
Pronounced recovery of ferrite during warm deformation and annealing	Plain medium carbon steel	0.22C-0.21Si-0.74Mn	1.3	1.6 at strain rate of 0.01 s ^{-1}	823	$823K\times120min$	[39]
Cold deformation and annealing of martensitic steel	Martensitic steel	0.13C-0.37Mn-0.01Si	0.18	0.8	293	$773 \text{ K} \times 30 \text{ min}$	[49]

Abbreviations: DRX^{*}, dynamic recrystallization; A_{r1}^* , austenite to pearlite transformation temperature during cooling; A_{c1}^* , pearlite to austenite transformation temperature during heating; ECAP, equal channel angular pressing; ARB, accumulative roll bonding; HPT, high pressure torsion; AC, air cooling; WC, water cooling; WQ, water quench.

mass production, but both approaches offer insight into the microstructure and properties that can be achieved by such approaches.

3. Microstructure characterization of ultrafine grained steels

Ultrafine grained ferrite microstructures can be quite different due to the various methods and heat treatments applied as well as the differences in the chemical compositions and the initial microstructures. In this section, characterization of ultrafine grained bcc steel microstructures will be discussed in detail.

3.1. Microstructure of ultrafine grained steels produced by SPD techniques

3.1.1. Equal-channel angular pressing

The microstructures of low carbon steels (0.15 wt.% C) after different passes of equal-channel angular pressing have been investigated by Fukuda et al. and Shin et al. [7,22]. After one ECAP pass (T = 623 K, $\varepsilon = 1.0$), the microstructure consisted of extended parallel grain boundaries with mainly low-angle misorientation angle between adjacent crystals [7,22]. The width of the parallel bands was approximately 0.3 µm and the dislocation density inside the subgrains was relatively low. After two ECAP passes ($\varepsilon = 2.0$), the average misorientation between subgrains increased, the ferrite grain shape was less elongated and the average grain size was approximate 0.5 µm. Equiaxed ferrite grains with an average grain size of 0.2-0.3 µm were achieved after four ($\varepsilon = 4.0$) ECAP passes. The fraction of high-angle grain boundaries increased gradually with further deformation passes. Consequently, the final microstructure of samples, which had undergone a sufficient number of ECAP passes consisted mainly of high-angle grain boundaries [7].

For a submicron grained low carbon steel processed by ECAP ($\varepsilon = 4.0$) at 623 K, less grain growth was observed at relatively low annealing temperatures (693–783 K for 1 h) [62]. Both the dislocation structure and the well-defined grain boundaries at elevated temperatures observed in the microstructure demonstrated the occurrence of recovery during annealing in this temperature region. A further increase in the annealing temperature (\geq 813 K) led to partial primary recrystallization. The addition of Ti and V to low carbon steels did not lead to significant refinement of ferrite after ECAP processing [59]. Nevertheless, very fine Ti–V nitrides were reported to be beneficial for improving work hardening of the steel by accumulation of dislocations around the precipitates.

It is well known that many ultrafine grained single phase steels exhibit relatively low tensile ductility at room temperature. This can be partially attributed to the low work hardening rate, which is commonly observed for ultrafine grained single phase material. One approach to improve the work hardening of such steels is to create microstructures, which contain a second phase. In this context, ultrafine grained dual phase steels seem to be attractive for obtaining both higher strength and improved ductility. Ultrafine grained ferrite–martensite dual phase steels (0.15% C) have been fabricated by Park et al. [48,63] using

ECAP plus intercritical annealing in the ferrite/austenite two phase region (i.e. between the A_{c1} and A_{c3} temperatures) followed by quenching. The microstructure of the steel after the ECAP deformation (T = 773 K, $\varepsilon = 4.0$) consisted of a severely deformed pearlitic lamellar microstructure with reduced interlamellar spacing, ultrafine ferrite with an average grain size of 0.2–0.5 µm with high dislocation density, and spheroidized cementite particles. After intercritical annealing at 1003 K for 10 min and subsequent water quenching, the microstructure consisted of ultrafine ferrite grains, homogeneously distributed martensite islands, and incomplete martensite networks at the ferrite-ferrite grain boundaries. The martensite islands were transformed from the austenite, which replaced pearlite during the intercritical annealing treatment. The martensite network was reported to be associated with local segregation of Mn [48,63]. High dislocation densities were observed in the ferrite grains adjacent to the martensite. Most of these dislocations were assumed to result from accommodation of the phase transformation during quenching. The high dislocation density enhanced the work hardening behavior. In summary, grain refinement was significant after the first pass of ECAP. A further increase in the number of deformation passes had a diminishing effect on grain refinement but was beneficial for the formation of high-angle grain boundaries and the transition of the ferrite grain morphology from an elongated to more equiaxed shape. The ultrafine grained microstructure produced by ECAP was relatively stable against grain coarsening at certain temperatures. Recovery was the main softening mechanism at modest annealing temperatures.

3.1.2. Accumulative roll bonding

Compared with the ultrafine grained microstructure produced by the other SPD and conventional rolling techniques, different types of microstructures and crystallographic textures were observed for steels produced by the ARB method [23,64,65]. This difference can be attributed to the different strain distributions associated with the various approaches. It is well known that the surface regions of ferritic steel sheets processed by large strain rolling reveal a pronounced shear texture which is quite different than the texture observed in the through-thickness center regions of the same sheet [66-69]. In the ARB technique, the rolled sheet is cut and stacked between ensuing cycles, so that half of the surface, which had undergone the severe shear deformation in the prior rolling step ends up in the sheet center in the following ARB rolling step. These shear regions appear not only at the surface layers, but are also distributed through the sheet thickness after several ARB passes. Materials processed by ARB undergo a complicated mixed series of plane strain and shear deformation states. Thus, steels processed by the ARB method experience a complex distribution of microstructure and texture through their sheet thickness [23,64,65].

Tsuji et al. [23,64] investigated the microstructure and crystallographic texture of an ultra-low carbon (0.003% C) IF steel processed by the ARB process. Experiments were conducted by imposing a logarithmic strain of $\varepsilon = 0.8$ (50% reduction) at 773 K. This procedure was repeated up to seven cycles corresponding to a total strain of 5.6. The microstructure after one cycle of the ARB process ($\varepsilon = 0.8$) showed a typical dislocation cell structure. The size and orientation of elongated cells varied through the sheet thickness. After two more cycles of the ARB process ($\varepsilon = 2.4$), elongated grains with high-angle misorientation were observed in addition to the dislocation cell structure. With further increases in strain ($\varepsilon \ge 3.2$) the resulting microstructure consisted mainly of elongated ultrafine ferrite grains, and an increased fraction of high-angle grain boundaries. After seven cycles of the ARB process ($\varepsilon = 5.6$) around 80% ultrafine ferrite grains were surrounded by high-angle grain boundaries, while some dislocations remained in the ferrite. The ultrafine grained microstructure was distributed relatively homogenously throughout the sheet thickness.

The ultrafine grained microstructure formed via the ARB process can be interpreted in terms of a process of repeated gradual recovery and grain subdivision. The extent of recovery is sufficient to result in high-angle grain boundaries after extensive ARB. The ARB method is more effective for achieving grain refinement than conventional routes at identical strains. The authors attributed this to the redundant shear strain throughout the thickness of specimens processed by the ARB, which facilitated grain subdivision and formation of an ultrafine grained microstructure [23,64,65].

3.1.3. High pressure torsion

The thickness reduction imposed on samples processed by HPT is negligible compared to the large shear strain imposed. The formation of nanostructures and the dissolution of pearlite lamella in a commercial pearlitic steel (~0.7% C) produced by HPT were reported by Ivanisenko et al. [16-18]. After a shear strain of 100 at room temperature the microstructure at the surface of a disk shaped sample consisted of a cell structure and partially dissolved cementite lamellae. Further increase in the shear strain to a level of 200 resulted in an inhomogeneous grain morphology. Elongated grains 100 nm in length and 15 nm in height were created during the process. The elongated grains were separated by dense dislocation walls. This morphology was very similar to the lamellar-type boundaries observed in samples processed by ECAP. The spacing of the cementite lamella decreased during straining. After a shear strain of 300, a homogeneous nanostructure with a grain size of 10 nm and total dissolution of cementite was obtained.

3.2. Microstructure of ultrafine grained steels produced by advanced thermomechanical processing

3.2.1. Transformation grain refinement

In low carbon microalloyed steels, ferrite grain sizes and precipitation states are important factors, which affect the strength-toughness relationship. The ferrite grain size is a function of the austenite grain size after austenite recrystallization, the amount of retained strain in the austenite before the start of transformation, and the cooling rate through the transformation regime [56].

Progressive refinement of the austenite can be achieved through dynamic and static recrystallization during large strain deformation (roughing) at temperatures above the recrystallization temperature. According to the work of Kaspar et al. [26], by strictly controlled hot deformation schedules, dynamic recrystallization of austenite is obtained at relatively low temperatures (less than 1143 K) by applying total finishing strains greater than 2.2 in a microalloyed steel (0.11C-0.34Si-1.45Mn-0.068Nb-0.08V, wt.%). The grain size of the dynamically recrystallized austenite was around $1-4 \,\mu m$. Priestner and Ibraheem [56] reported that fine austenite with grain size of $<5 \,\mu$ m could be obtained by reheating a cold-rolled tempered martensite (with finely dispersed cementite) in a Nb microalloyed steel (0.1C-0.31Si-1.42Mn-0.035Nb, wt.%) [56]. Average ferrite grain sizes of $<1 \,\mu$ m in the surface layer of a 2-3 mm thick sheet have been achieved using accelerated cooling (e.g. $\sim 8 \text{ K s}^{-1}$) after hot rolling of fine austenite to equivalent strains of 0.5–1.0 at 1123 K [56]. The ferrite grain size in the center of the plate was $\sim 1.5 \,\mu$ m. Studies using EBSD and misorientation imaging showed that most of the grain boundaries revealed misorientations above 15° [56].

Contrary to the accepted view that fine austenite grain sizes lead to fine ferrite grains, Hurley and Hodgson [54] showed that a very fine ferrite grain size could be produced from a steel having a large prior austenite grain size. Intragranular nucleation of ferrite may be an important factor contributing to the additional grain refinement observed when a dynamic strain-induced transformation occurs, and is encouraged by large austenite grain sizes and accelerated cooling, both of which suppress the formation of grain boundary proeutectoid ferrite [54]. The straininduced transformation rolling procedure is attractive in terms of its relative simplicity and ability to refine ferrite grain sizes in plain carbon steels [70-75]. The technique involves rolling steel strip containing a large austenite grain size (>100 µm), at a temperature just above the A_{r3} but below the A_{e3} . A single rolling pass induces very efficient grain refinement, producing equiaxed and fine polygonal ferrite grains on the scale of less than 2 μ m in the surface regions (~250 μ m deep) of the strip [53]. The rolling reduction required to generate this ultrafine ferrite is approximately 35-40%. It appears that a roll chilling effect in conjunction with large shear strains resulting from roll friction explain the phenomenon. These steps facilitate a high density of intergranularly nucleated ferrite grains during hot rolling of austenite.

Using large strain ($\varepsilon = 2.3$) hot rolling in the austenite/ferrite two-phase region, followed by fast cooling, Nanba et al. [60] produced ultrafine ferrite with a grain size of 1.2 µm in a low alloyed steel (0.17C-0.44Si-1.32Mn-0.015Nb, wt.%). In contrast, Bodin et al. [76] reported that a bimodal grain size distribution was obtained by hot rolling in the two-phase region. Conceivably, the large ferrite grains (>6 µm in diameter) observed in the bimodal size distribution can be attributed to growth of the transformed ferrite into the deformed ferrite. The transformed ferrite resulted from austenite that was deformed during intercritical rolling, while the deformed ferrite was transformed from austenite before intercritical rolling. The small ferrite grains (1-2 µm in diameter) were attributed to extended recovery of the deformed ferrite [76]. In order to obtain homogeneous ultrafine ferrite by intercritical rolling, it seems to be very important to balance the dynamic transformation of austenite into ferrite and the dynamic

recovery and recrystallization of ferrite through careful control of the processing parameters including chemical composition, deformation schedules (strain/strain rate/temperature), and cooling rate. For example, low carbon steels have a relatively small intercritical regime and recrystallization of deformed ferrite can proceed rapidly but is terminated upon rapid cooling.

3.2.2. Grain refinement by recovery/recrystallization in warm working

Since hot working involves a high cost of thermal energy, there has been a trend to develop processes at lower temperatures [77]. Deformation at lower temperature, also referred to as warm working, can help to produce steels close to their final shape and reduce or eliminate cold work involving higher roll forces or die-pressures. Grain refinement during warm or ferritic rolling can be realized by recovery/recrystallization. In this context, dynamic recrystallization of ferrite under conditions of temperature and strain rate that correspond to a large Zener–Hollomon parameter, i.e. at low temperatures and high strain rates, is more beneficial to obtain good microstructure homogeneity.

In contrast to the accepted view that grain refinement is achieved by recrystallization, Song et al. [40,44] have recently proposed that pronounced or extended recovery is more effective for the formation of ultrafine microstructure. In their studies, the prevalence of primary recrystallization instead of recovery was not generally beneficial since it significantly reduced the dislocation density and removed the substructure that was important for the gradual formation of subgrains and of ultrafine grains surrounded by HAGBs.

3.2.3. Grain refinement by cold deformation and annealing

It is known that the grain size obtained by static recrystallization is a function of the prior strain and the prior grain size [56]. Cold rolling and annealing of an initial martensite microstructure have drawn some attention recently to produce multiphase ultrafine grained steels [49–51]. The initial fine martensite is beneficial for grain subdivision during cold rolling due to the high dislocation density and substantial amount of solute carbon atoms in martensite. Nearly equiaxed ferrite grains and a homogeneous distribution of carbides were found after annealing. A multiphase ultrafine grained steel, consisting of ultrafine ferrite, dispersed cementite and tempered martensite, showed a good combination of strength and ductility.

3.3. Summary: production of ultrafine grained microstructures

In order to more quantitatively evaluate the microstructure of ultrafine grained steels, it has become customary to report not only the average cell or grain sizes and the corresponding grain size distributions, but also the fraction of high-angle grain boundaries obtained from the various processing strategies. The submicron structure produced by SPD is typically more elongated due to the intense deformation involved. Around 40% of the grain boundaries are of the low-angle dislocation boundary type (misorientations < 15°), which is less beneficial for the overall mechanical response. These low-angle grain boundaries often appear in TEM as dense dislocation walls, rather than as sharp boundaries, which could migrate more easily. It is difficult for the cells to be transformed into discrete grains surrounded by high-angle grain boundaries without an annealing treatment. The conversion to high-angle misorientation walls usually occurs at a temperature of $0.3-0.4T_{\rm M}$ (melting temperature), which is much below the traditional static recrystallization temperature of $0.5T_{\rm M}$ [61].

Hot deformation develops larger more polygonized cells or subgrains during dynamic recovery compared to the submicron structure produced by SPD. Increasing strain leads to the occurrence of dynamic recrystallization of austenite. Hot working at intermediate temperature often provides a mixed microstructure of different grain sizes. Warm and cold working hastens grain subdivision due to a relatively higher dislocation density introduced/accumulated compared to hot deformation. Subsequent annealing is beneficial for formation of high-angle grain boundaries by pronounced recovery/recrystallization processes.

The effects of alloying are largely similar in the different types of processing. Solid solution additions usually increase the degree of strain hardening in both cold and hot working and may slow dynamic recovery in bcc steels. Large quantities of second phase constituents, such as fine cementite particles, are beneficial for the formation of a fine ferritic grain structure. They inhibit grain boundary migration due to Zener pinning. This effect stabilizes the ultrafine grains against grain coarsening, and is also thought to inhibit primary recrystallization. The presence of such fine particles results in an increase of the effective recrystallization temperature, widening the temperature windows for corresponding warm rolling and annealing treatments [39].

4. Tensile properties

4.1. Strength

4.1.1. Effect of grain size on strength

The yield stress for bcc steels processed by different methods is plotted in Fig. 2 as a function of the inverse square root of the grain size for grain sizes ranging from 45 to 0.2 µm. The ultrafine microstructures (grain size less than 2 µm) were produced by various techniques: the open symbols display the results from the SPD methods; the full symbols in gray represent the results from the advanced thermomechanical process routes (ATP); the full symbols in black show the results from the conventional route (Conv). For each class of steel, the yield stress follows the Hall–Petch relation for a given steel, $\sigma_y = \sigma_i + k_y d^{-1/2}$, where σ_y is the yield stress, σ_i the friction stress, k_y the grain boundary resistance and *d* is the grain size in µm.

The lower yield strength of the 0.13C–0.67Mn–0.14Si (wt.%) steel sheet produced by cold rolling and annealing [82] is shown by the solid diamond in Fig. 2 where the grain size varied from 1.6 to 30 μ m. The friction stress σ_i is about 100 MPa and the grain boundary resistance k_y is 551 MPa μ m^{1/2} [82], according to the work of Morrison in 1966.

ECAP (at 623 K) followed by annealing at temperatures between 373 and 873 K produced steels with grain sizes rang-



Fig. 2. Hall–Petch relationship in ultrafine grained bcc steels [7,46,48, 59,78–82]. The open symbols display the results from the SPD methods; the full symbols in gray represent the results from the advanced thermomechanical process routes (ATP); the full symbols in black show the results from the conventional route (Conv). The straight lines show the Hall–Petch relation for different steels.

ing from about 0.23 to 10 μ m in a low carbon (0.15C–1.1Mn–0.25Si, wt.%) and a low alloy steel (0.15C–1.1Mn–0.25Si–0.06V, wt.%) [78]. The k_y value in Fig. 2 (slope of bold line) is smaller in the steel processed by ECAP compared with the results of Morrison (dashed bold line). The yield stress for a grain size of 30 μ m before ECAP is above the value predicted by Morrison, while the yield stress after ECAP is below the line. This phenomenon also reappears in other studies from both SPD and advanced thermomechanical processes [7,46,48,59,79–81]. That is, while the Hall–Petch relationship in steels may extend to the submicron range, the parameter k_y may decrease. The reason for this behavior will be discussed in Section 4.1.3.

For steels with submicron grain sizes produced by ECAP, the yield stress for steels with a carbon content less than 0.1 wt.% [7,59] is notably smaller than for the steels with 0.15 wt.% carbon [78] for a given grain size. The reason for this behavior is not fully understood, but could result from differences in grain size measurement.

The data for samples with a dual phase microstructure (displayed by the sun symbol in Fig. 2) [46] do not follow the line predicted by the Hall–Petch relationship as mentioned above. It seems that a smaller increment in stress is achieved in the dual phase steel when the ferrite grain size is refined from 19.4 to $0.8 \,\mu\text{m}$. It is not clear whether this is related to some variation in the amount and morphology of the second phase after grain refinement.

4.1.2. Summary of Hall–Petch analysis for bcc steels

It should be stressed that in early investigations by Morrison [82], as shown in Fig. 2, the different grain sizes were produced by cold rolling and subsequent annealing at different temperatures. This offered the advantage to alter only one parameter—the grain size. In the investigation by Song et al. (where the initial motivation was not to measure the value of k_y and σ_i in the Hall–Petch equation), the coarse microstructure consisted of conventional ferrite and pearlite. When refined into the ultrafine microstructure, however, it comprised ferrite and fine spheroidized cementite. A smaller k_y value was found by Shin et al. [79], which might also be attributed to the change in overall microstructure (along with grain size) in their study. By use of the ECAP technique, the initial coarse grained ferrite–pearlite microstructure was severely deformed. After four deformation passes, a microstructure with finer ferrite and a partially spheroidized pearlite was obtained. Thus, the smaller k_y value in some studies on ultrafine ferrite might be the result of a reduction in the yield strength by replacing harder pearlite with softer ferrite and spheroidized cementite in the ultrafine microstructure. The presence of low misorientations between some grains in the ultrafine ferrite may also contribute to the reduced k_y value in comparison to conventional "coarse" ferrite with high misorientations.

It should be mentioned that most of the submicron microstructures measured for the SPD technique consist of large quantities of low-angle grain boundaries, and grain dimensions measured refer to the thickness of stretched microbands, which is not the same as average grain diameter. Further consideration of grain morphologies and appropriate characterization methods may be worthwhile to define the Hall–Petch relationship more accurately.

4.1.3. Comments on the effect of ultra grain refinement on the Hall–Petch k_y value

A series of early experimental investigations using Armco iron and nickel [83,84] over a broad range of grain size showed that the Hall–Petch relationship was an approximation applicable only over a limited range of grain sizes. The value of k_y seems to decrease for very small grain sizes. This deviation of the Hall–Petch relationship has been noted since the late 1950s and early 1960s [85–88]. Efforts have been made to develop an understanding of this behavior.

For polycrystalline materials, there exist three main theories for the Hall–Petch equation: the pile-up models [86,89–91], those based on work hardening [88,92,93] and the grain boundary source theories [94,95]. Pande et al. [96] demonstrated that the decrease of k_y at small grain sizes can be explained within the framework of the traditional dislocation pile-up model. The solution of the pile-up problem for small numbers of dislocations (n < 20) differs considerably from the usual solution [97] valid for larger n. With smaller grain sizes the $\sigma_y(d^{-1/2})$ relationship becomes a staircase function that reaches a plateau equal to $\sigma_y^{max} = M\tau_c$ at n = 1, where M is the Taylor factor and τ_c is the critical shear stress required for dislocation motion.

Fig. 3 shows a comparison of calculated exact and approximate n values together with the Hall–Petch prediction. It can be observed from Fig. 3 that the linear Hall–Petch relation is valid for this model when n > 20. If the length of one pile-up is assumed to be equal to half of the grain diameter, L, when n is equal to 20, the grain size/diameter is about 0.79 µm. This means based on the prediction in Fig. 3 a smaller k_y value results when the grain size is less than 0.79 µm. According to the results from SPD as displayed in Fig. 2, k_y maintains the same value when grain size is varies from 10 to 0.23 µm for a given steel. Therefore, it can be concluded that the smaller value of k_y in the present study is not fully explained by the model discussed above.



Fig. 3. Comparison of the exact and approximate *n* value (number of dislocations) together with the Hall–Petch prediction. After [96]. The exact value is calculated from the data of [98]. The approximate curves exhibit discrete steps and begin to level off as described by [99]. *L* is the length of the pile-up which is associated with the grain size, *b* the magnitude of Burger's vector, σ the applied stress and σ^* is the barrier stress which is assumed to be constant and independent of grain size; μ is the shear modulus.

Recent studies have reduced grain sizes to a few nanometers. Compared to conventional polycrystalline materials, nanocrystalline materials have often been found to exhibit a smaller or even a negative Hall–Petch slope. The critical grain size where deviation from Hall–Petch relation occurs is dependent on the specific material of interest [100].

4.2. Ductility

Several groups [27,40,52,101,102] have reported promising room temperature tensile strength properties for ultrafine grained steels. The steels are produced either by the severe plastic deformation or by the advanced thermomechanical processes. Many of the ultrafine grained steels investigated do not display a significant amount of work hardening, however. This shortcoming is reflected in high yield ratios (lower yield stress to ultimate tensile stress). For many ultrafine grained steels, the yield ratios are almost 1.0, compared to 0.7 for conventional steels with similar alloy content.

Reduced work hardening typically leads to low tensile ductility in ultrafine grained steels. According to the work of Park et al. [102], an ultrafine grained low carbon steel (0.15C–1.1Mn–0.25Si, wt.%) with a grain size of 0.2 μ m, manufactured by severe plastic deformation (accumulative equivalent strain of 4.0 at 623 K), exhibited no work hardening, i.e. necking occurred already in the Lüders regime. Therefore, only a small "uniform" elongation was reported. As an example, Fig. 4 provides data on tensile ductility versus inverse square root of grain size for bcc steels with grain sizes of 150–0.2 μ m. For each of the steels, the total elongation is represented by an open symbol and the uniform elongation is displayed by a filled symbol. The figure shows that a decrease in grain size leads to a decrease in ductility. A sudden drop of elongation at a grain size of about 1 μ m was reported in the study by Tsuji et al. (circles) for an



Fig. 4. Grain size dependence of ductility for bcc steels [24,44,46,102–105]. Open symbols represent total elongation while filled symbols display uniform elongation in tension.

IF steel refined by the ARB process at 773 K and subsequent annealing [24]. It is interesting to note that this tendency does not apply to the ultrafine grained dual phase steel (diamonds) according to Son et al. [46], produced by ECAP with an effective strain of around 4.0 at 773 K and subsequent intercritical annealing at 1003 K for 10 min. The uniform elongation was higher for the ultrafine grained dual phase steel (the sizes of the ferrite grains and martensite islands were about $0.8 \,\mu$ m), while the total elongation was comparable to its coarse grained counterpart, having ferrite grain and martensite island diameters of about 19.4 and 9.8 μ m, respectively. The authors attributed the better ductility in the ultrafine grained dual phase steel to extensive work hardening associated with a high density of mobile dislocations.

The decrease in tensile ductility at room temperature for most of the ultrafine grained steels, especially single phase steels, can be explained as follows. First, dynamic recovery as a softening mechanism is able to reduce the apparent work hardening rate. During deformation, dislocations that carry the intragranular strain are trapped at grain boundaries. The kinetics of dynamic recovery are associated with the spreading of trapped lattice dislocations into grain boundaries especially in ultrafine grained steels [106–108]. The change of the dislocation density during dynamic recovery in terms of the trapped lattice dislocations spreading into the grain boundaries was studied in detail by Park et al. [102]. The authors calculated approximate recovery times for dislocations moving into grain boundaries, and showed that for ultrafine grained steels the time for dislocations moving into grain boundaries is shorter than the time of the tensile test. This decrease in dislocation density reduces accumulation of dislocations inside grains, and consequently leads to less work hardening when compared with corresponding steels of large grain size. Following these earlier investigations, it is suggested that there are two kinds of recovery mechanisms, namely, slow recovery in the grain interiors and much faster recovery in the vicinity of grain boundaries. In coarse grained steels, the latter mechanism is less important due to the lower volume fraction of material near grain boundaries. Taking the study of Song et al. [44] for example, a plain carbon steel (0.2 wt.% C) grain diameter was reduced from 6.8 to 1.3 μ m. This grain refinement enhanced the fraction of the overall volume near grain boundaries by a factor of about 143. Thus, in ultrafine grained steels, faster recovery near grain boundaries seems to be important.

Second, the decrease in tensile ductility can be explained in terms of plastic instability, which initiates necking due to localized deformation. The condition for initiation of necking in a uniaxial tensile test is indicated by the Considère criterion [109], $\sigma_t = d\sigma_t/d\varepsilon_t$. When the slope of the true-stress true-strain curve (work hardening rate), $d\sigma_t/d\varepsilon_t$, is equal to the true stress, σ_t , uniform deformation stops and necking is initiated. As mentioned above, ultra grain-refinement greatly increases the flow stress of steels, especially during the early stages of plastic deformation. Grain refinement also leads to reduced work hardening capacity. As a result, plastic instability (necking) occurs at an early stage during tensile testing, which results in limited uniform elongation in ultrafine grained steels.

The yield ratio is high in ultrafine grained steels. However, according to the study by Song et al. [44], good ductility can still be obtained in 0.2% C steel, as documented by a total elongation of about 20% and uniform elongation of about 10% (Fig. 5). These values differ from the results reported in previous studies, where total elongations are usually below 10%. The high ductility observed by Song et al. was attributed to the presence of finely dispersed cementite particles, which increase the work hardening rate [42]. A large volume fraction and a fine dispersion of cementite effectively increase the work hardening rate by promoting accumulation of dislocations around the particles [110,111]. Another approach to improve the tensile ductility of ultrafine grained steel at room temperature is to adopt a composite structure in which only the surface is ultrafine, while the core with a coarse microstructure provides ductility. An interesting extension of this idea is to employ ultrafine grains locally, only where they are needed in the product to locally generate high strength and toughness [112].



Fig. 5. Comparison of engineering stress–strain curves of the 0.2% C steels with different ferrite grain sizes. The different grain sizes were produced by the conventional route (without large strain warm deformation) and the ultrafine grain route, respectively. The ultrafine grain route involved a warm deformation procedure with four steps (each deformation step with $\varepsilon = 0.4$ and $\dot{\varepsilon} = 10 \text{ s}^{-1}$) and a subsequent 2 h annealing treatment at 823 K. The symbol d_{α} refers to the average ferrite grain diameter. After Song et al. [44].

4.3. Lüders strain

It is well known that a decrease in grain size leads to an increase in Lüders strain as illustrated in Fig. 5 [44]. A large Lüders strain has also been noted by Lloyd and Morris [113] in a fine grained $(1-3 \mu m)$ Al-6% Ni alloy that contained small amounts of magnesium in solid solution. They observed that the reduction of grain size entailed an increase in yield stress and a decrease in work hardening. Hayes and Wang [114,115] conducted a study on the influence of grain refinement on Lüders strain in Al alloys. They investigated the serrated strain regime for specimens with various grain sizes between 0.4 and 20 µm and observed that the Lüders strain was linearly proportional to the inverse square root of the grain size in Al alloys, as in the Hall-Petch relationship. The appearance of pronounced yield drops and very large Lüders strain regimes thus appear to be characteristics of ultrafine grained Al alloys as well as steels [44,114]. These phenomena can be linked to an instantaneous low density of mobile dislocations, lack of dislocation sources within grains, and the low work hardening rate of ultrafine grained alloys.

The serrated flow that characterizes the propagation of plastic strain within a Lüders band is governed by the dynamic interplay of micromechanical hardening and softening. The Lüders regime is determined by the population of mobile dislocations, the strain hardening coefficient, the strain softening coefficient, the strain rate and temperature [42]. Song et al. [42] reported that yielding involved the initiation of deformation bands due to local stress concentrations. Owing to the high density of mobile dislocations formed by unlocking and by dislocation multiplication, the material within the deformation band effectively softens and undergoes localized plastic deformation. As mentioned in Section 4.2, dynamic recovery is pronounced in steels with smaller grain sizes owing to fast recovery in the vicinity of grain boundaries [102]. A decrease in the work hardening rate in the ultrafine grained steel, which can be attributed to the rapid dynamic recovery, favors a non-uniform deformation mode like local deformation by Lüders bands. This leads to slow propagation of the Lüders band front in the steel with a fine microstructure. The slow propagation is coupled with a large Lüders strain.

5. Toughness of ultrafine grained bcc steels

5.1. Toughness improvement in ultrafine grained steels

While several studies examined tensile properties of ultrafine grained steels, Charpy impact properties were less commonly investigated due to limitations in the sample size typically available from laboratory-scale process set-ups.

The impact properties of ultrafine grained IF, low/medium carbon and Nb–V–Ti microalloyed steels have been reported by Tsuji et al. [116], Hanamura et al. [105], Song et al. [44] and Sjong et al. [117]. Fig. 6 shows the impact transition curves of the medium carbon steels (0.2 wt.% C) for subsize ($3 \text{ mm} \times 4 \text{ mm}$) specimens [44]. Compared with conventional steel (grain size: 6.8μ m), the upper shelf energy is lower and the transition


Fig. 6. Dependence of the Charpy impact properties on temperature of the steels with different ferrite grain sizes [44]. The symbol d_{α} refers to average ferrite grain diameter. DBTT_{subsize}: ductile-to-brittle transition temperature of subsize specimen with a 1 mm notch depth and a ligament size of 3 mm × 3 mm. The ductile-to-brittle transition temperature was determined by using the correlation procedure recommended in [118].

region occurs over a wider temperature range in the ultrafine grained steel (grain size: $1.3 \,\mu$ m). The ductile-to-brittle transition temperature was defined as the temperature at half of the upper shelf energy [44]. Fig. 6 shows the decrease in ductile-to-brittle transition temperature (from 193 to 153 K) associated with grain refinement into the ultrafine ferrite regime. In the ductile-to-brittle transition region, the temperature dependence

of the absorbed energy is reduced for the ultrafine grained steel. Currently, there is insufficient data to report quantitatively on the relationship between grain size and toughness in the ultrafine and nanocrystalline regime.

5.2. Fundamental explanation for the low ductile-to-brittle transition temperature in ultrafine grained steels

5.2.1. Effect of grain refinement on improving toughness

A reduction in the average grain size commonly leads to a lower ductile-to-brittle transition temperature. This can be understood in terms of cleavage crack initiation and propagation. It is known that the grain size is one of the major factors determining the cleavage fracture unit [119,120]. A decrease in grain size can limit the propagation of initiated cleavage cracks and raise the fracture toughness in the transition region. Since the ductile-to-brittle transition temperature is the point at which the yield stress is equal to the cleavage fracture stress, the ductileto-brittle transition temperature is lowered by grain refinement due to a more significant increase in the cleavage fracture stress than in the yield stress.

5.2.2. *Effect of delamination on lowing the ductile-to-brittle transition temperature*

Delamination behavior in Charpy specimens has been reported by several researchers [44,116,121–124]. As shown in Fig. 7, a decrease in grain size or Charpy impact testing temperature leads to an increase in the number of delaminations.



Fig. 7. Scanning electron microscope (SEM) images of fracture surfaces for the steels with different ferrite grain sizes after subsize $(3 \text{ mm} \times 4 \text{ mm})$ Charpy V-notch impact tests. (a) Fracture surface of the conventional 0.2% C steel (average ferrite grain diameter of 6.8 µm) after impact testing at 293 K; (b–d) fracture surfaces of the ultrafine grained 0.2% C steel (average ferrite grain diameter of 1.3 µm) after impact testing at 293, 233 and 103 K, respectively. The black arrows point out material delaminations. After [44].

The origin of the delaminations is not completely understood at present. From previous studies it seems that distorted ferrite-pearlite microstructures [125], elongated ferrite grain shapes [121], certain texture characteristics [44,124,126], and aligned particles and inclusions [44,127] favor the occurrence of delamination. However, the phenomenon of delamination does not have a direct influence on the speed of crack growth in ductile failure [128]. Nevertheless, delamination leads to a reduction of the ductile-to-brittle transition temperature in the impact test samples of the ultrafine grained steel due to a decrease in the triaxiality of the stress state [44].

5.3. Shelf energy

The ductile-to-brittle transition in steels is associated with two different failure mechanisms. At high temperatures in the upper shelf region, fracture occurs by nucleation and coalescence of microvoids entailing ductile tearing. This process requires extensive plastic deformation and large amounts of energy. At low temperatures, fracture occurs by cleavage, which is the sudden separation of atomic planes across the specimen [125,129]. In this case, less energy is required.

5.3.1. Lower shelf energy

Fig. 6 shows that the lower shelf energy is significantly higher in the ultrafine grained steel than in the coarse grained steel. On the one hand, this can be attributed to the effect of grain refinement on improving toughness even at very low temperatures. This behavior is shown by the presence of about 50% shear fracture in the ultrafine grained subsize specimen when the test temperature was as low as 103 K. Low temperature toughness can also be enhanced by anisotropic microstructure or pronounced crystallographic texture of the ultrafine grained steel produced by the large strain deformation below the A_1 temperature (austenite to pearlite transformation finish temperature) [44].

Fig. 8a shows the fracture surface of an ultrafine grained 0.2% C steel after Charpy impact testing at 103 K. The highmagnification view of the fracture surface in Fig. 8b clearly shows the smooth delamination surface as well as the dimpled ductile fracture area. The smooth undulating surface suggests some type of decohesion of the grain boundaries. Fig. 8c shows delaminations in the rolling direction, and Fig. 8d shows delaminations following the elongated grain boundaries. The occurrence of delamination along the grain boundaries, both above and below an elongated grain, indicates that the crack can make minor adjustments in its propagation direction switching from one grain boundary to another. This is also confirmed by the observation that two elongated grains (i.e. grain "1" and grain "2" in Fig. 8d) with different texture components, $\langle 1 1 1 \rangle \parallel ND$ and $(001) \parallel$ ND, respectively, were separated by a crack. The delaminations appear to propagate by means of a low-energy fracture mechanism that produces a fairly smooth fracture surface. This fracture does not exhibit the typical cleavage appear-



Fig. 8. SEM micrographs and ND (normal direction) orientation map (taken by electron backscatter diffraction (EBSD) measurement) of ultrafine grained 0.2% C steel (average ferrite grain diameter of 1.3 μ m) after subsize Charpy impact testing at 103 K. The images shown in (a) and (b) are taken from a plane normal to the rolling direction (RD), while (c) and (d) are normal to the transverse direction (TD) of the sample. Orientation components in (d), $\langle 1 1 1 \rangle \parallel$ ND in blue, $\langle 0 0 1 \rangle \parallel$ ND in red and $\langle 1 0 1 \rangle \parallel$ ND in green. After [44]. (a) Overall fracture surface; (b) transition between delaminated and shear fracture regions. The observation area of (b) is shown in (a). (c) Longitudinal cross-section. The black arrows point out chains of large voids in the specimen; (d) crack propagation along interfaces. The circles 1 and 2 show two elongated grains with high-angle grain boundaries in between. The observation area of (d) is shown in (c).

ance with a strong (100) texture [121] and contributes to a higher fracture toughness in the lower shelf energy region for the ultrafine grained steel investigated due to reduced triaxiality of the stress state [44]. According to Song et al. [44], a high-angle grain boundary can act as a favorable path for crack propagation especially when large cementite particles are located along the boundary. An alternating microstructure of ferrite and aligned cementite can facilitate the spread of cracks in both the transverse and rolling directions.

5.3.2. Upper shelf energy

A reduced upper shelf energy can be observed in Fig. 6 in the ultrafine grained steel compared with a conventional steel of the same composition. This may be due to the relatively low work hardening and ductility of this steel, consistent with the smaller integrated area below the engineering stress–strain curve, Fig. 5. Delaminations in the ultrafine grained steel observed in the upper shelf region may also contribute to the reduced upper shelf energy.

Compared to the shear fracture surface in the conventional steel (Fig. 7a), a few delaminations can be observed in the ultrafine grained steel (Fig. 7b) Charpy impact specimen tested at room temperature (in the upper shelf region). Since there is little plastic deformation in the area of the delamination, lower absorbed energy and reduced fracture toughness is not surprising in the ultrafine grained steel (Fig. 7b) compared with the conventional steel exhibiting complete shear fracture (Fig. 7a). This differs from the lower shelf energy region, where a decrease in the ductile-to-brittle transition temperature is evident in the ultrafine grained steel due to the change in the stress triaxiality associated with relaxing σ_{zz} . As a result, reduced upper shelf energy and reduced ductile-to-brittle transition temperature appear characteristic of ultrafine grained steel processed by large strain warm deformation (Fig. 6).

According to the work of Fujioka et al. [130], a reduction of the upper shelf energy was also observed in a 0.16C-0.44Si-1.33Mn-0.012Ti-0.013Nb steel with a grain size of 1.5 µm. The ultrafine grained steel in their study was produced by flat rolling with a total logarithmic strain of $\varepsilon \approx 2.5$ at 973 K. They attributed the reduced value of the upper shelf energy to the elongated grain morphology in the ultrafine grained steel. However, in the study of Nagai [131] the same value of upper shelf energy was found for an ultrafine and a coarse grained steel (0.15C-0.3Si-1.5Mn) with grain sizes of 0.9 and 20 µm, respectively. The ultrafine grained steel was fabricated by warm rolling. The sample was rotated 90° about the rolling direction after each pass in order to conduct multi-directional deformation. The upper shelf energy was unexpectedly high, and was explained by the low impurity level of the steel investigated [131].

According to reports in the literature on the shelf energy of ultrafine grained steels [44,130,131], a reduced value of the upper shelf energy in two-phase ultrafine grained materials may be mainly due to the anisotropic microstructure resulting from the large strain deformation. Currently, large strain deformation at a low deformation temperature is a favorable method to produce ultrafine grained microstructures. Therefore, it might be particularly attractive in the future to develop ultrafine grained steels by the use of relatively lower strains and higher temperatures to develop microstructures with fewer delaminations.

6. Conclusions

Processing, microstructure and mechanical properties of ultrafine grained bcc steels were discussed and compared with several of their coarse grained counterparts. The following conclusions can be drawn based on the interpretations presented in this paper:

- (1) Ultrafine grained bcc steels can be produced by severe plastic deformation techniques or advanced thermomechanical processing routes. For the severe plastic deformation methods, a well-designed strain path is more important and also more feasible than a precisely controlled temperature path. The small scale, complexity and the discontinuous nature of these processes suggest that they would require considerable ingenuity and investment to be applied on a high-volume industrial scale. Compared with severe plastic deformation methods, the advanced thermomechanical processing routes are less effective with respect to grain refinement, but they are more efficient with respect to large sample sizes. A further difference between these two approaches is that the advanced thermomechanical methods are continuous processes, require less total strain, and can be readily optimized when they work in a temperature regime where they exploit phase transformation.
- (2) The submicron structure produced by SPD is typically more elongated due to the intense deformation involved. Around 40% of the grain boundaries are usually subgrain boundaries (grain boundary misorientations <15°) so that many cells are not actually grains but subgrains which are less beneficial for the overall mechanical response of such specimens. It is difficult for the cells to be transformed into real grains, which are surrounded by high-angle grain boundaries without an annealing treatment.

Hot deformation develops larger more polygonized cells or subgrains as a result of dynamic recovery. Hot working at intermediate temperature often provides a mixed microstructure. Deformation induced grain subdivision is essential for the formation of ultrafine grained microstructures by warm and cold working. Pronounced recovery/recrystallization processes are necessary to form highangle grain boundaries.

(3) An improved combination of strength and toughness is obtained in ultrafine grained steels compared with their coarse grained counterparts. Reasonable ductility in ultrafine grained steel can be attributed to the presence of finely dispersed particles which improve the work hardening capacity owing to the accumulation of geometrically necessary dislocations around the particles. Ultrafine grained steel exhibits a large Lüders strain because of the relatively low work hardening rate due to rapid dynamic recovery in ultrafine grained steel compared with coarse grained steel. In ultrafine grained steel, the upper shelf energy is relatively low due to the occurrence of delaminations. Some factors such as crystallographic texture and alignment of cementite particles along the ferrite grain boundaries, etc., may promote the formation of delaminations. The lower shelf energy is significantly raised and the ductile-to-brittle transition temperature is reduced in ultrafine grained steel compared to conventional steel. This can be attributed to the joint effect of the small ferrite grain size and the occurrence of delamination, which involves a decrease in the triaxiality of the stress state in the impact test samples of the ultrafine grained steel.

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References

- [1] G. Frommeyer, J.A. Jiménez, Metall. Mater. Trans. A 36 (2005) 295.
- [2] R.Z. Valiev, A.V. Sergueeva, A.K. Mukherjee, Scripta Mater. 49 (2003) 669.
- [3] I.V. Alexandrov, R.Z. Valiev, Scripta Mater. 44 (2001) 1605.
- [4] V.V. Stolyarov, Y.T. Zhu, T.C. Lowe, R.K. Islamgaliev, R.Z. Valiev, Nanostruct. Mater. 11 (1999) 947.
- [5] R.Z. Valiev, Mater. Sci. Eng. A 234 (1997) 59.
- [6] V.M. Segal, Mater. Sci. Eng. A 271 (1999) 322.
- [7] Y. Fukuda, K. Oh-Ishi, Z. Horita, T.G. Langdon, Acta Mater. 50 (2002) 1359.
- [8] J. Kim, I. Kim, D.H. Shin, Scripta Mater. 45 (2001) 421.
- [9] D.H. Shin, I. Kim, J. Kim, K.T. Park, Acta Mater. 49 (2001) 1285.
- [10] Z. Horita, M. Furukawa, M. Nemoto, T.G. Langdon, Mater. Sci. Technol. 16 (2000) 1239.
- [11] Y.T. Zhu, T.C. Lowe, Mater. Sci. Eng. A 291 (2000) 46.
- [12] Y. Saito, H. Utsunomiya, N. Tsuji, T. Sakai, Acta Mater. 47 (1999) 579.
- [13] N. Tsuji, R. Ueji, Y. Minamino, Scripta Mater. 47 (2002) 69.
- [14] N. Tsuji, Y. Saito, H. Utsunomiya, S. Tanigawa, Scripta Mater. 40 (1999) 795.
- [15] T. Inoue, S. Torizuka, K. Nagai, Proceedings of the International Symposium on Ultrafine Grained Steels (ISUGS 2001), The Iron and Steel Institute of Japan, Fukuoka, Japan, 2001, pp. 88–91.
- [16] Y. Ivanisenko, R.Z. Valiev, W. Lojkowski, A. Grob, H.J. Fecht, Ultrafine Grained Materials, vol. II, TMS, Seattle, USA, 2002, pp. 47–54.
- [17] Y. Ivanisenko, R.K. Wunderlich, R.Z. Valiev, H.J. Fecht, Scripta Mater. 49 (2003) 947.
- [18] Y. Ivanisenko, W. Lojkowski, R.Z. Valiev, H.J. Fecht, Acta Mater. 51 (2003) 5555.
- [19] R.Z. Valiev, I.V. Alexandrov, Ann. Chim. Sci. Matér. 27 (2002) 3.
- [20] V.M. Segal, V.I. Reznikov, A.D. Drobyshevskiy, V.I. Kopylov, Russ. Metall. 1 (1981) 99.
- [21] R.Z. Valiev, N.A. Krasilnikov, N.K. Tsenev, Mater. Sci. Eng. A 137 (1991) 35.
- [22] D.H. Shin, B.C. Kim, Y.-S. Kim, K.-T. Park, Acta Mater. 48 (2000) 2247.
- [23] N. Kamikawa, N. Tsuji, Y. Minamino, Sci. Technol. Adv. Mater. 5 (2004) 163.
- [24] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino, Scripta Mater. 47 (2002) 893.
- [25] Y. Saito, N. Tsuji, H. Utsunomiya, T. Sakai, R.G. Hong, Scripta Mater. 39 (1998) 1221.
- [26] R. Kaspar, J.S. Distl, O. Pawelski, Steel Res. 59 (1988) 421.

- [27] P.D. Hodgson, M.R. Hickson, R.K. Gibbs, Scripta Mater. 40 (1999) 1179.
- [28] H. Mabuchi, T. Hasegawa, T. Ishikawa, ISIJ Int. 39 (1999) 477.
- [29] Z.M. Yang, R.Z. Wang, ISIJ Int. 43 (2003) 761.
- [30] J.K. Choi, D.H. Seo, J.S. Lee, K.K. UM, W.Y. Choo, ISIJ Int. 43 (2003) 764.
- [31] M.R. Hickson, P.J. Hurley, R.K. Gibbs, G.L. Kelly, P.D. Hodgson, Metall. Mater. Trans. A 33 (2002) 1019.
- [32] S.C. Hong, S.H. Lim, K.J. Lee, D.H. Shin, K.S. Lee, ISIJ Int. 43 (2003) 394.
- [33] Y. Matsumura, H. Yada, ISIJ Int. 27 (1987) 492.
- [34] A. Najafi-Zadeh, J.J. Jonas, S. Yue, Metall. Trans. A 23 (1992) 2607.
- [35] N. Tsuji, Y. Matsubara, Y. Saito, Scripta Mater. 37 (1997) 477.
- [36] S.V.S. Murty, S. Torizuka, K. Nagai, T. Kitai, Y. Kogo, Scripta Mater. 53 (2005) 763.
- [37] J. Baczynski, J.J. Jonas, Metall. Trans. A 29 (1998) 447.
- [38] Y. Weng, ISIJ Int. 43 (2003) 1675.
- [39] R. Song, D. Ponge, D. Raabe, R. Kaspar, Acta Mater. 53 (2005) 845.
- [40] R. Song, R. Kaspar, D. Ponge, D. Raabe, Ultrafine Grained Materials, vol. III, TMS, Charlotte, North Carolina, USA, 2004, p. 445.
- [41] R. Song, D. Ponge, R. Kaspar, D. Raabe, Z. Metallkd. 95 (2004) 513.
- [42] R. Song, D. Ponge, D. Raabe, Scripta Mater. 52 (2005) 1075.
- [43] R. Song, D. Ponge, R. Kaspar, Steel Res. 75 (2004) 33.
- [44] R. Song, D. Ponge, D. Raabe, Acta Mater. 53 (2005) 4881.
- [45] R. Song, D. Ponge, D. Raabe, ISIJ Int. 45 (2005) 1721.
- [46] Y.I. Son, Y.K. Lee, K.-T. Park, C.S. Lee, D.H. Shin, Acta Mater. 53 (2005) 3125.
- [47] G. Azevedo, R. Barbosa, E.V. Pereloma, D.B. Santos, Mater. Sci. Eng. A 402 (2005) 98.
- [48] K.-T. Park, S.Y. Han, B.D. Ahn, D.H. Shin, Y.K. Lee, K.K. Um, Scripta Mater. 51 (2004) 909.
- [49] R. Ueji, N. Tsuji, Y. Minamino, Y. Koizumi, Acta Mater. 50 (2002) 4177.
- [50] N. Tsuji, R. Ueji, Y. Minamino, Y. Saito, Scripta Mater. 46 (2002) 305.
- [51] R. Ueji, N. Tsuji, Y. Minamino, Y. Koizumi, Sci. Technol. Adv. Mater. 5 (2004) 153.
- [52] M.R. Hickson, P.D. Hodgson, Mater. Sci. Technol. 15 (1999) 85.
- [53] P.J. Hurley, P.D. Hodgson, B.C. Muddle, Scripta Mater. 40 (1999) 433.
- [54] P.J. Hurley, P.D. Hodgson, Mater. Sci. Eng. A 302 (2001) 206.
- [55] H. Mabuchi, T. Hasegawa, T. Ishikawa, ISIJ Int. 39 (1999) 477.
- [56] R. Priestner, A.K. Ibraheem, Mater. Sci. Technol. 16 (2000) 1267.
- [57] H. Yada, Y. Matsumura, K. Nakajima, United State Patent, 4,466,842, Nippon Steel Corporation, Tokyo (1984).
- [58] D.J. Hamre, D.K. Matlock, J.G. Speer, in: E. Essadiqi, J. Thomson (Eds.), Ultra-fine Structured Steels, Canada Institute of Mining, Metallurgy, and Petroleum, Montreal, Canada, 2004, pp. 109–121.
- [59] W.J. Kim, J.K. Kim, W.Y. Choo, S.I. Hong, J.D. Lee, Mater. Lett. 51 (2001) 177.
- [60] S. Nanba, M. Nomura, N. Matsukura, K. Makii, Y. Shirota, Proceedings of the International Symposium on Ultrafine Grained Steels (ISUGS 2001), The Iron and Steel Institute of Japan, Fukuoka, Japan, 2001, pp. 286– 289.
- [61] H.J. McQueen, N.D. Ryan, in: E. Essadiqi, J. Thomson (Eds.), Ultra-fine Structured Steels, Met. Soc. CIM, 2004, pp. 55–67.
- [62] D.H. Shin, B.C. Kim, K.-T. Park, W.Y. Choo, Acta Mater. 48 (2000) 3245.
- [63] K.-T. Park, Y.K. Lee, D.H. Shin, ISIJ Int. 45 (2005) 750.
- [64] N. Tsuji, Y. Saito, S.-H. Lee, Y. Minamino, Adv. Eng. Mater. 5 (2003) 338.
- [65] X. Huang, N. Tsuji, N. Hansen, Y. Minamino, Mater. Sci. Eng. A 340 (2003) 265.
- [66] M. Hölscher, D. Raabe, K. Lücke, Steel Res. 62 (1991) 567.
- [67] M. Hölscher, D. Raabe, K. Lücke, Acta Metall. 42 (1994) 879.
- [68] D. Raabe, K. Lücke, Mater. Sci. Technol. 9 (1993) 302.
- [69] D. Raabe, Mater. Sci. Technol. 11 (1995) 461.
- [70] M.R. Hickson, R.K. Gibbs, P.D. Hodgson, ISIJ Int. 39 (1999) 1176.
- [71] Z.M. Yang, R.Z. Wang, ISIJ Int. 43 (2003) 761.
- [72] J.-K. Choi, D.-H. Seo, J.-S. Lee, K.-K. Um, W.-Y. Choo, ISIJ Int. 43 (2003) 746.
- [73] P.J. Hurley, G.L. Kelly, P.D. Hodgson, Mater. Sci. Technol. 16 (2000) 1273.

- [74] S.C. Hong, S.H. Lim, K.J. Lee, D.H. Shin, K.S. Lee, ISIJ Int. 43 (2003) 394.
- [75] S.C. Hong, K.S. Lee, Mater. Sci. Eng. A 323 (2002) 148.
- [76] A. Bodin, J. Sietsma, S. van der Zwaag, Mater. Charact. 47 (2001) 187.
- [77] G.H. Akbari, C.M. Sellars, J.A. Whiteman, Acta Mater. 45 (1997) 5047.
- [78] D.H. Shin, J.-J. Park, S.Y. Chang, Y.-K. Lee, K.-T. Park, ISIJ Int. 42 (2002) 1490.
- [79] D.H. Shin, C.W. Seo, J. Kim, K.-T. Park, W.Y. Choo, Scripta Mater. 42 (2000) 695.
- [80] M.Y. Liu, B. Shi, C. Wang, S.K. Ji, X. Cai, H.W. Song, Mater. Lett. 57 (2003) 2798.
- [81] R. Song, Microstructure and Mechanical Properties of Ultrafine Grained C–Mn Steels, Shaker Verlag GmbH, Aachen, 2005, p. 126.
- [82] W.B. Morrison, Trans. ASM 59 (1966) 824.
- [83] A.A.W. Thompson, Acta Metall. 23 (1975) 1337.
- [84] A.A.W. Thompson, Acta Metall. 25 (1977) 83.
- [85] W.M. Baldwin, Acta Metall. 6 (1958) 139.
- [86] R.W. Armstrong, I. Codd, R.M. Douthwaite, N.J. Petch, Philos. Mag. 7 (1962) 45.
- [87] J.D. Campbell, K.J. Marsh, Philos. Mag. 7 (1962) 933.
- [88] H. Conrad, Acta Metall. 11 (1963) 75.
- [89] N.J. Petch, Iron Steel Inst. 174 (1953) 25.
- [90] A.H. Cottrell, Trans. Metall. Soc. AIME 212 (1958) 192.
- [91] J.S. Koehler, Phys. Rev. 85 (1952) 480.
- [92] A.A. Johnson, Philos. Mag. 7 (1962) 177.
- [93] H. Conrad, S. Feuerstein, L. Rice, Mater. Sci. Eng. 2 (1967) 157.
- [94] J.C.M. Li, Trans. Metall. Soc. AIME 227 (1963) 239.
- [95] J.C.M. Li, Y.T. Chou, Metall. Trans. 1 (1970) 1145.
- [96] C.S. Pande, R.A. Masumura, R.W. Armstrong, Nanostruct. Mater. 2 (1993) 323.
- [97] J.D. Eshelby, F.C. Frank, Philos. Mag. 42 (1951) 351.
- [98] T.E. Mitchell, S.S. Hecker, R.L. Smialek, Phys. Stat. Sol. 11 (1965) 585.
- [99] R.W. Armstrong, Y.T. Chou, R.M. Fisher, N. Louat, Philos. Mag. 14 (1966) 943.
- [100] H.W. Song, S.R. Guo, Z.Q. Hu, Nanostruct. Mater. 11 (1999) 203.
- [101] M. Liu, B. Shi, G. Bi, H. Cao, X. Cai, H. Song, Mater. Sci. Eng. A 360 (2003) 101.
- [102] K.T. Park, Y.S. Kim, J.G. Lee, D.H. Shin, Mater. Sci. Eng. A 293 (2000) 165.
- [103] A. Azushima, K. Aoki, Mater. Sci. Eng. A 337 (2002) 45.
- [104] K.-T. Park, S.Y. Han, D.H. Shin, Y.-K. Lee, K.J. Lee, K.S. Lee, ISIJ Int. 44 (2004) 1057.

- [105] T. Hanamura, F. Yin, K. Nagai, ISIJ Int. 44 (2004) 610.
- [106] R.Z. Valiev, E.V. Kozlov, Y.F. Ivanov, J. Lian, A.A. Nazarov, B. Baudelet, Acta Metall. Mater. 42 (1994) 2467.
- [107] J. Lian, B. Baudelet, A.A. Nazarov, Mater. Sci. Eng. A 172 (1993) 23.
- [108] W. Lojkowski, Acta Metall. Mater. 39 (1991) 1891.
- [109] R.E. Reed-Hill, R. Abbaschian, Physical Metall Principles, PWS Publishing Company, 1994, p. 158.
- [110] M.F. Ashby, Philos. Mag. 21 (1970) 399.
- [111] J.C. Fisher, E.W. Hart, R.H. Pry, Acta Mater. 1 (1953) 336.
- [112] A.A. Howe, Mater. Sci. Technol. 16 (2000) 1264.
- [113] D.J. Lloyd, L.R. Morris, Acta Metall. 25 (1977) 857.
- [114] J.S. Hayes, R. Keyte, P.B. Prangnell, Mater. Sci. Technol. 16 (2000) 1259.
- [115] Z.C. Wang, P.B. Prangnell, Mater. Sci. Eng. A 328 (2002) 87.
- [116] N. Tsuji, S. Okuno, Y. Koizumi, Y. Minamino, Mater. Trans. 45 (2004) 2272.
- [117] A.L. Sjong, R.L. Bodnar, M.J. Merwin, D.K. Matlock, J.G. Speer, in: R.I. Asfahani, R.L. Bodnar, M.J. Merwin (Eds.), Proceedings of the Symposium on Microalloyed Steels, Ohio, 2002, pp. 92–100.
- [118] R. Kaspar, H. Faul, MP Materialprüfung 43 (2001) 18.
- [119] S. Kim, Y.R. Im, S. Lee, H.C. Lee, Y.J. Oh, J.H. Hong, Metall. Mater. Trans. A: Phys. Metall. Mater. Sci. 32 (2001) 903.
- [120] P. Brozzo, G. Buzzichelli, A. Mascanzoni, M. Mirabile, Met. Sci. 11 (1977) 123.
- [121] B.L. Bramfitt, A.R. Marder, Metall. Trans. A 8 (1977) 1263.
- [122] T. Hashimoto, T. Sawamura, H. Ohtani, Iron Steel Inst. Jpn. 65 (1979) 1425.
- [123] S. Matsuda, Y. Kawashima, S. Sekiguchi, M. Okamoto, Iron Steel Inst. Jpn. 68 (1982) 436.
- [124] A. Ohmori, S. Torizuka, K. Nagai, Iron Steel Inst. Jpn. 89 (2003) 765.
- [125] P. Shanmugam, S.D. Pathak, Eng. Fract. Mech. 53 (1996) 991.
- [126] R. Schofield, G. Roentree, N.V. Sarma, R.T. Weiner, Met. Technol. (1974) 325.
- [127] A.J. McEvely, R.H. Rush, Trans. ASM 55 (1962) 654.
- [128] E. Miyoshi, M. Fukuda, H. Iwanaga, Okazawa T, Proceedings of the Inst. of Gas Eng. Conference, 1974 (paper 4).
- [129] W. Pitsch, G. Sauthoff, H.P. Hougardy, in: V.D. Eisenhüttenleute (Ed.), A Handbook for Mater Research and Engineering, vol. 1, Springer-Verlag, 1992, p. 278.
- [130] M. Fujioka, T. Yokota, Y. Adachi, N. Matsukura, Proceedings of the Second Symposium on Super Metal, Japan, 1999, p. 193.
- [131] K. Nagai, Mater. Proc. Technol. 117 (2001) 329.