ULTRAFINE GRAINED STEELS BY ADVANCED THERMOMECHANICAL PROCESSES AND SEVERE PLASTIC DEFORMATIONS

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ABSTRACT: On the wake of the advances in Australia and Japan in achieving even finer grain sizes, the European steel companies became particularly interested in the prospects for 1 μm-UFF. For this reason, under the auspices of the European Community for Steel and Coal (ECSC), a preliminary one-year feasibility study was commissioned in 1999 to assess the properties and prospects for bulk manufacture of fine (sub-micron/micron) structures and starting from the results of this project, another project was commissioned.
During this new research, different refining mechanisms and innovative deformation cycles applied to a wide range of chemical compositions were studied. The primary objective of the project was to optimise processing routes for 2-3 μm grained mixed microstructures in the bulk or surface layers of strip, plate or rods. The metallurgical aspects were focused on processing routes that do not require extreme strains and significant plant changes, through the study of microalloying additions when necessary.
This paper summarizes the results from this new project regarding both ultrafine surface grained steels, obtained exploiting the Deformation Induced Ferrite Transformation mechanism (DIFT), and the Accumulative Roll Bonding technique.

KEY WORDS: High Strength Steel, ultrafine grain, DIFT, Deformation Induced Ferrite Transformation, Accumulative Roll Bonding, ultrafine surface steels.

INTRODUCTION

On the wake of the advances in Australia and Japan in achieving even finer grain sizes, the European steel companies became particularly interested in the prospects for 1 μm-UFF. Under the auspices of the ECSC, in 1999 a one-year feasibility study was therefore commissioned under Howe [1], involving British Steel (now Corus) and Manchester University/UMIST MMSC, Belgium (CRM), Germany (RWTH-Aachen) and Italy (CSM), to investigate the production and properties of ultra-fine grained steels and to identify if this was indeed a potential major commercial opportunity, a potential niche-market product, or indeed just an academic curiosity.
This project concluded that such microstructures were achievable commercially, although requiring significant investment on conventional industrial plants. However, such fine sizes did not appear actually to be desirable in terms of properties combination (high strength but poor plasticity and little work hardening). Thus, it was concluded that both single phase and even dual phase material would probably benefit from having slightly less fine grain sizes (1-3 μm).
Starting from this experience, a 3-years project [2] was commissioned in 2001 to assess the properties and prospects for bulk manufacture of fine (sub-micron/micron) structures. The work was extended from typical lean strip compositions to ultra-high carbon steels, and included both the definition of advanced thermomechanical processes that can be applied on already existent pilot mills or that can be applied off line (Pony Mill). The use of different severe plastic deformation techniques such as Equal Channel Angular Extrusion and Accumulative Roll Bonding was also considered.
The grain size range aimed by this project was 1-3 μm that represents the best compromise between a useful strength increment over conventional products and the reduction of production costs, while maintaining an adequate stability of mechanical properties during the production

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cycle. The formation of UFF combined with other small microstructural constituents (e.g. cementite, martensite islands) should maintain enough work hardening characteristics.

The advantages of UFG microstructures were mainly assessed for the following types of steels:
Low carbon: 0.04-0.16C; 0.15-0.3Si; 0.5-1.6Mn
Medium carbon:0.25-0.45C;0.15-0.3Si; 0.8-1.3Mn.
High carbon: from 0.8% up to 1.6 %.C
The benefits of micro-additions of Nb (0.04-0.1%) and/or other elements (e.g. P) were examined.
The project was divided into 4 work packages (WPs) which are briefly discussed.

WP1. UF Surface Grained Steel
The objective of this work package was to obtain ultra fine surface grained steels from plain and microalloyed low C steels (0.05-0.1%C). The method of producing a UFF surface layers in thin strip exploited the deformation induced ferrite transformation mechanism (DIFT).

WP2. UF Low C Steel
The objective of this work package was to investigate the processing routes for the production of a fine grained ferritic steel with a grain size < 2 μm, having improved mechanical properties in comparison with existing industrially produced HSLA steel. Effect of Nb, Mn and Si in solution on $\gamma \rightarrow \alpha$ transformation kinetics was assessed.

WP3. UF Medium C Steel
The objective of this work package was to define suitable thermo-mechanical processing conditions to produce ultra-fine grain microstructures in plain medium C with 0.15-0.3% carbon, consisting of ultrafine ferrite grains and uniformly distributed cementite particles, based on the Pony Mill concept.

WP4. UF High C Steel
The aim of this work package was to define suitable thermo-mechanical processing conditions to produce ultra-fine grain microstructures in high carbon steels (0.6 – 1.6 wt. %).

As an alternative to conventional rolling and advanced thermomechanical processing, the potential to produce ultra-fine microstructures by severe plastic deformation technique like Equal Channel Angular Extrusion (ECAE) and Accumulative Roll Bonding was also evaluated.

This project was a multi-partner collaboration between CSM (Italy), CRM (Belgium), MPIE (Germany) and CORUS (UK) and was coordinated by CSM.
In this paper, results from an European project on advanced thermomechanical process (exploiting DIFT mechanism) and Accumulative Roll Bonding are summarized.

STRAIN INDUCED DYNAMIC TRANSFORMATION AND ULTRAFINE SURFACE GRAINED STEEL

One of the objectives of this project was to obtain ultra fine surface grained steels for plain and microalloyed low-C steels (0.05-0.1%C).
The possibility of making microstructures in which bands of Ultra Fine Ferrite (UFF) grains act as strengthening component to the standard microstructural constituents was investigated in thin strips characterised by very large austenite grains by Hodgson et al. [3,4].

The layered microstructure consisting of ultra fine grained surface layers (generally penetrating to one quarter of the strip thickness) in the plates were found to be beneficial for improving the YS to UTS ratio.
Their method of producing a surface layer of UFF in thin strip required austenitization at a high temperature to form a coarse austenite grain. The strip was then cooled and hot rolled at a temperature close to the Ar_3 value (700-800°C), using a modest reduction (30-40%). The undercooling of austenite owing to roll chilling, combined with the strong shear strain at the strip surface, lead to a very high nucleation density of ferrite on dislocations within the coarse austenite grains. It seemed that the UFF grains resulted from strain-induced transformation of intragranular ferrite with nucleation occurring on the austenite substructure. The reduction or minimisation of grain boundary nucleation by austenite grain enlargement seemed to promote a substantially instantaneous transformation homogeneously over the austenite. This process is called Deformation Induced Ferrite Transformation (DIFT).
A lot of studies have been carried out in recent years to well understand DIFT mechanism and to characterize it [5-11].
It was concluded that it is a dynamic phase transformation occurring during deformation at temperature slightly higher than Ar_3 due to the strain energy accumulated in the austenite phase that induces an early $\gamma-\alpha$ phase transformation.

The aim of this experimentation was first to assess the hot deformation conditions in the austenite region for producing fine grain sizes through DIFT mechanism, then, application of these results to production of ultrafine surface grained steels was carried out.

Experimental

Deformation dilatometry was used to determine the necessary process parameters for the different steel compositions. Experiments were mainly carried out to assess the hot deformation conditions for producing fine grain sizes through DIFT mechanism and to establish the influence of chemical composition, prior austenitic grain size, strain and deformation temperature on grain refinement. After the determination of DIFT parameters, laboratory rolling tests were carried out to reproduce as closely as possible the deformation schedules used in dilatometer experiments in order to get ultra fine grains on sheets surface.
The chemical compositions of the steels used for these tests and the measured critical temperatures are given in Tab.1.

	C	Si	Mn	P	S	Al	N	Ar_1	Ar_3
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	°C	°C
A	0.047	0.2	1.44	0.007	0.0023	0.0259	0.0028	625	823
B	0.107	0.2	1.45	0.007	0.0021	0.0258	0.0032	610	790

Table 1- Chemical compositions and critical temperatures measured by dilatometric tests.

In addition, two microalloyed steels having the same C content and 0.03%Nb were used to verify the influence of Nb on DIFT occurrence.
The hot deformation experiments were performed by a Theta Ind. Dilatronic III dilatometer. Samples had 3.25 mm diameter and 6 mm length. The experiments were carried out in vacuum ($2x10^{-5}$ mbar) and quenching was done using He. The cooling rate was 130°C/s.

Results

The study of the effect of different parameters (prior austenite grain size, strain, deformation temperature, carbon content) on DIFT mechanism were carried out.
In order to get different prior austenite grain sizes before deformation, the samples were heated at different temperatures and different times, then cooled to the deformation temperature of about Ar_3+25°C and deformed giving 40% and 60% reductions (Fig. 1). After deformation, the samples were helium-quenched. Samples for metallographic examination were prepared from the deformed specimens by polishing the longitudinal section and observed at the centre of the sample.
In Fig. 2 the microstructures of sample B are shown.
In Fig. 2a, the microstructure of the sample having a small prior austenitic grain size (13μm), 40% deformed at 816 °C is reported. Ferrite that nucleated at the austenite grain boundaries can be clearly seen. When the reduction was increased to 60% (Fig. 2c) the microstructure consisted of predominantly fine ferritic grains and small amount of bainite and martensite. The average ferrite grain size (FGS) was 3 μm.
In the case of larger prior austenitic grain size (133 μm) and 40% reduction (Fig. 2b), no polygonal ferrite was detected. If the reduction is increased to 60% the microstructure consisted of some fine ferritic grains at the austenite boundaries, Widmanstätten ferrite (WF) and bainite. The average ferritic grain size in this case was 3.6 μm (Fig. 2d).

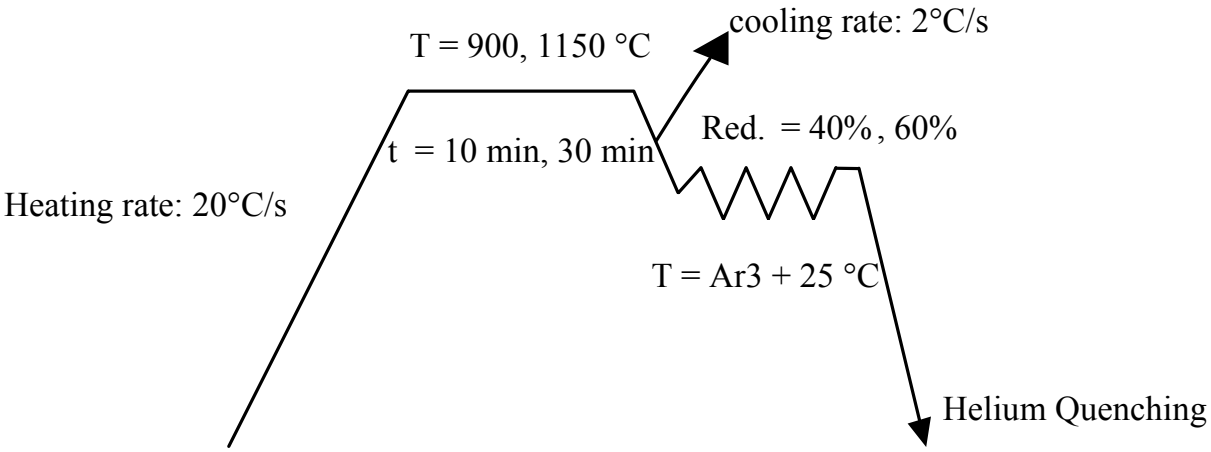


Fig. 1 – Thermo-mechanical cycles used for determination of DIFT parameters.

The very fine polygonal ferrite grains observed in quenched specimens after deformation were formed during deformation. In fact, results from preliminary experimentation showed that the ferrite could not be formed without deformation in the later stages of cooling. The presence of ferrite in the samples showed in Fig. 2 means that the γ–α transformation occurred dynamically during deformation. However, the steel A showed some pro-eutectoid ferrite in the quenched samples due to its lower hardenability.
In Fig. 3 the microstructures of the steel A deformed at 837 °C are shown.

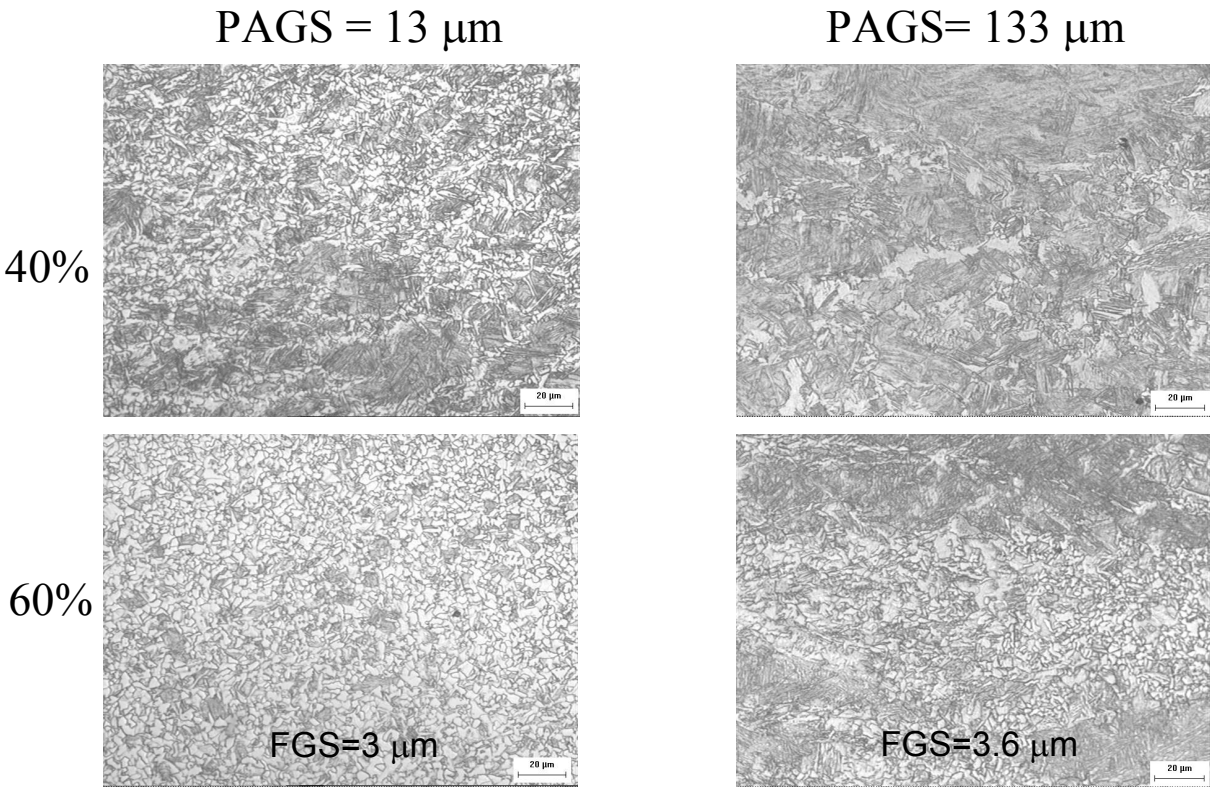


Fig. 2-Microstructures of steel B (0.1%C) after quenching, for different prior austenitic grain size and reductions.

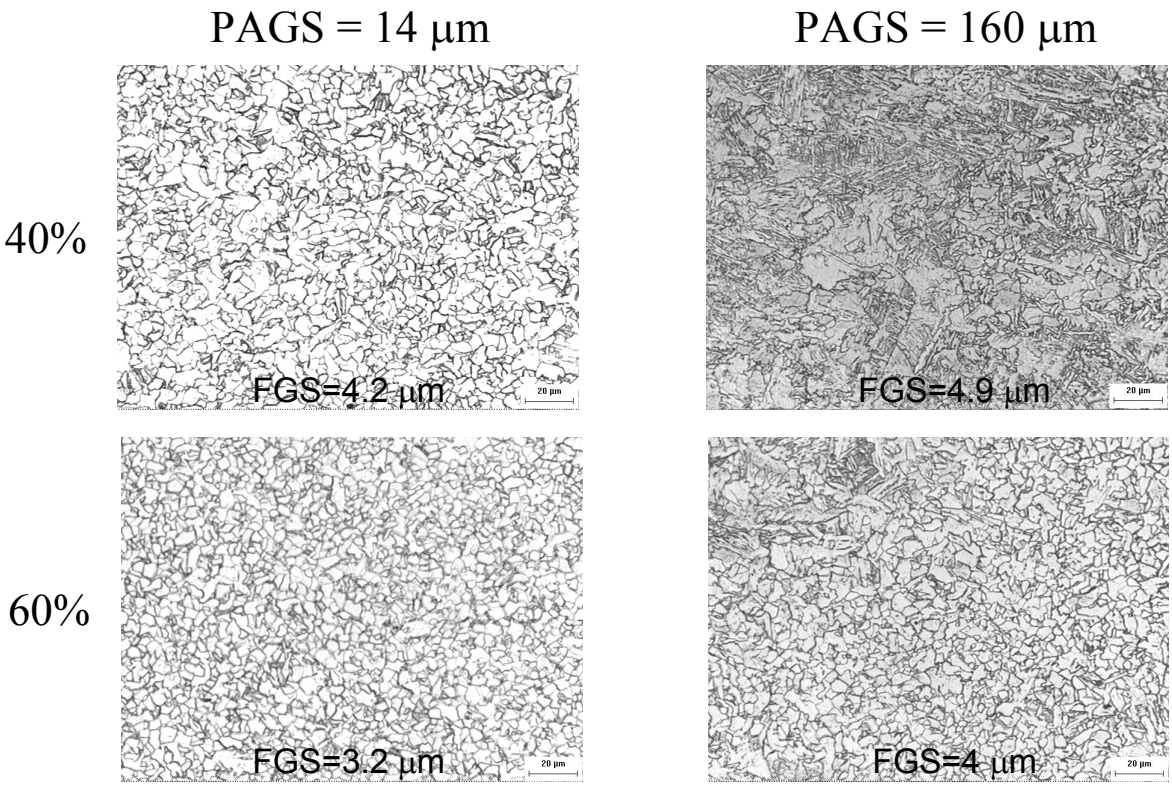


Fig. 3– Microstructure of steel A after quenching for different prior austenitic grain size and reductions.

The microstructure of samples strained to 40% reduction with small prior austenite grain size (14 μm) showed predominantly polygonal ferrite with a small amount of bainite. No WF or

degenerate ferrite was observed. In this sample, in addition to strain induced ferrite, there was some pro-eutectoid ferrite. This was evident from the two sets of grain sizes present in the deformed samples. The average grain size was 4.2 μm.
At 60 % deformation the microstructure was fully ferritic and the average grain size was 3.2 μm. In this case higher DIFT ferrite volume fraction was detected.
In the case of the sample with coarse prior austenite grain size (160 μm) deformed 40% the microstructure consisted of some fine ferritic grains at the austenite boundaries, Widmanstätten ferrite and bainite. At a higher reduction (60%) most of the structure consisted of polygonal ferrite but some bainite and WF ferrite were still present. The average ferritic grain size in this case was 4 μm. It is evident that a critical strain for DIFT occurrence exists and that depends on carbon content and prior austenite grain size.

Tests regarding the effect of deformation temperature on the occurrence of DIFT were carried out. Two tests were performed giving 40% reduction at two different deformation temperatures: the first specimen was deformed at Ar_3+15 °C and the other one at Ar_3+35 °C in order to verify some differences in grain size and ferrite volume fraction.
Results showed that grain size decreases with the decrease of the deformation temperature, and the ferrite volume fraction increases. It means that the critical strain for DIFT is decreased with the reduction of temperature.

Finally, similar dilatometric tests were carried out on samples having the same carbon content of the samples A and B but microalloyed with 0.03%Nb. in order to asses the influence of Nb content on DIFT mechanism occurrence. The results showed that Nb increases the strain necessary for DIFT occurrence.

Discussion

It is evident from the results that the processing parameters and the initial microstructure influence the occurrence of DIFT, the final grain size of ferrite and the DIFT ferrite volume fraction. They will be discussed hereinafter in more detail:

Critical strain
Deformation accelerates the phase transformation kinetics. However if the imposed strain is too small the dynamic transformation is not activated. Therefore, it is clear that there is a critical strain required for DIFT to occur and it is related to the deformation conditions and chemical composition.
From the above results the increase in the content of carbon in solution increases the critical strain retarding the DIFT mechanism, while smaller prior austenite grain sizes shows the smaller critical strain value.
In addition, even the deformation temperature has an important influence on critical strain. It was found that the critical strain required for DIFT to occur decreases with the deformation temperature, due to the increase of the transformation driving force.

Strain
It can be confirmed that increasing strain, more and finer DIFT grains can be obtained. When the strain is small, strain induced transformed ferrite starts to form along the prior austenite grain boundaries. As the strain is increased, the interiors of austenite grains are activated as nucleation sites for DIFT ferrite, which are probably deformation bands or twin boundaries.
Results found in literature show that the relationship between DIFT final grain size and strain shows an asymptotic trend increasing the strain. In fact it can be observed from the above results

that increasing reduction from 40% to 60% the ferrite grain size decreases only of 20% (e.g. from 4.2 μm to 3.2 μm), that is in fully agreement with the other results [6,8,10].
It means that increasing and increasing the strain, grain size does not decrease significantly.

Prior austenite grain size

Smaller prior austenitic grain size, the higher is the volume fraction of DIFT ferrite under a given deformation condition. This is mainly because the prior austenite grain boundary is a preferential nucleation site for DIFT ferrite.
A smaller prior austenite grain size has the longer grain boundary area in a given volume of material, and this will result in the larger volume fraction of ferrite. Moreover, a finer austenite grain reduces the critical strain for DIFT and therefore results in a finer ferrite grain size at a given strain.

Deformation Temperature

Deformation temperature influences the critical strain, grain size and ferrite volume fraction. the lower the deformation temperature, the lower the critical strain. If the critical strain for DIFT is reduced, a finer ferrite grain size and a higher volume fraction at a given strain are observed.
The effect of deformation temperature is important when the strain is relatively small, because a decrease of the deformation temperature is very effective in increasing the amount of DIFT. On the other hand, a decrease of the deformation temperature when relatively high strains are applied affects only slightly the amount of DIFT.

Steel chemistry

The carbon content has an important role on the formation of ultrafine grains. The level of ferrite refinement increases slightly when the carbon content increases but also the critical strain increases.
Moreover, the presence of Nb retards the DIFT occurrence in terms of higher critical strain needed to trigger the DIFT mechanism.
Therefore, results show that DIFT was inhibited in the presence of niobium in solid solution compared to a niobium-free steel. However, in the case the precipitates of Nb are not dissolved during heating preceding deformation, DIFT occurs in the Nb-steel in a very similar manner as in the Nb-free steel, except that the final structure is finer [9].

Strain Rate

In this experimentation strain rate was not considered. It is generally thought that the rate of deformation has a small or even negligible effect on refining grain size during DIFT. A strain rate in the range of 0.1-10 s^{-1} was not found to affect significantly the grain size [11].
However Mintz et al. [18] showed that the strain rate can have a certain influence on the fraction of DIFT ferrite especially when it is formed from a coarse austenite grain. If the strain rate is very low, then extensive dynamic recovery occurs, but if the strain rate is higher, the work hardening takes place and the conditions for DIFT occurrence are verified. For a finer austenite grain size the strain rate has less influence on the ferrite grain size and the fraction of DIFT.
For a given strain a higher strain rate and a lower deformation temperature are beneficial for the formation of DIFT ferrite [10].

LABORATORY ROLLING

The results by deformation dilatometry were applied to pilot hot rolling mill in order to develop ultrafine surface grained steels.
DIFT route was applied and surface grains whose size was around 4-5 μm for plain low C steels and 2-3 μm for the microalloyed one were detected.
High shear strain at the surface and low deformation temperature are the main factors which promote the formation of UFF surface layers. The smaller the prior austenite grains, the finer the DIFT ferrite grain. By exceeding the critical strain, the volume fraction of ferrite is increased.
It is evident that the chemical composition of the steel influences the morphology and the volume fraction of ultrafine grains formed at the surface layers of a strip and it significantly changes the microstructure formed in the core.
A special note must be done regarding the influence of cooling path following deformation.
Cooling applied just after rolling is a common practice in order to limit the growth of ferrite and so refine the final structure. This should be also true in the case of strain induced ferrite, and the role of the cooling path following deformation is of major interest to be investigated.
Besides, cooling also controls the type and morphology of second phases, which have a direct influence on the final strain hardenability of the steels.
However, in the industrial plants there exists a delay between rolling and cooling, that will also affect the microstructure, since it enhances the growth of existing ferritic nuclei. Therefore the effect of static recrystallization must be considered.

ACCUMULATIVE ROLL BONDING

Accumulative Roll Bonding (ARB) [12-15] is a severe plastic deformation technique able to impose a large deformation on massive samples at pilot scale. In ARB a strip is placed on the top of another strip, stacked together and rolled (Fig. 4). The resulting length of the material is then sectioned into two halves that are again stacked together and rolled. The whole process can be repeated again and again to accumulate strain. In ARB, rolling is not only a deformation process but also a bonding process carried out to obtain one-body solid final material.
In order to achieve a good bonding, the surfaces of the strips are cleaned both mechanically and chemically. Before rolling often the strips are heated before rolling to facilitate bonding and to reduce the rolling force. In such case the heating temperature must be below the recrystallization temperature of the material because recrystallisation cancels out the accumulated strain.
In this paper, severe plastic deformation was applied to a plain low C steel by ARB process. The samples were warm rolled at 615 °C with reduction of 50% per pass and the process was repeated 5 times in order to have a total strain of $\varepsilon=4$.
The resulting microstructure, texture and misorientation distribution of ARB specimens were studied.

Experimental

Trial experiments were carried out using a 0.15% C-1.37% Mn commercial steel strip. The initial size of samples was 300 mm in length, 50 mm in width and 3.7 mm in thickness. After degreasing and wire-brushing the surfaces to remove the oxide scale, two strips were overlapped, edge welded, heated and roll bonded by 50% reduction in thickness ($\varepsilon=0.8$) in one pass.
Initial technological difficulties were encountered in the production of ARB samples. The main problems were: inadequate bonding due to oxidation at the interface, soaking time and rolling

temperature optimisation in order to prevent recrystallisation and to accumulate strain after each pass.

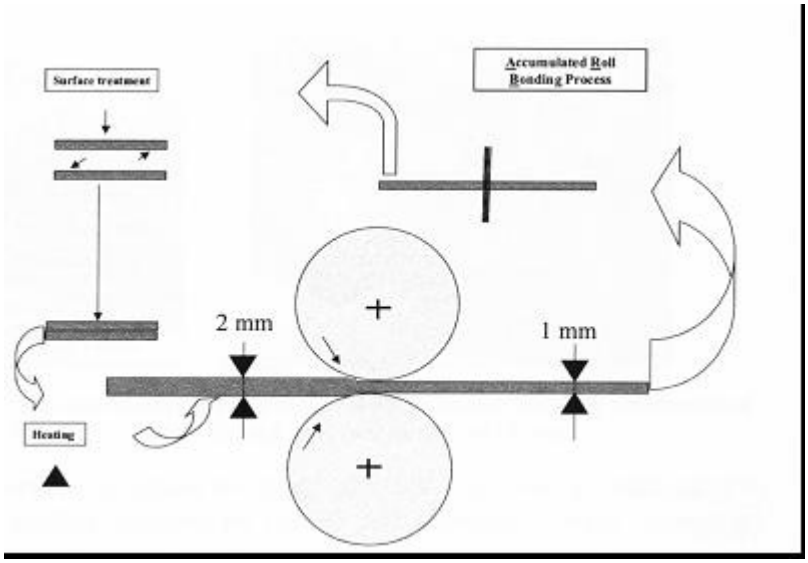
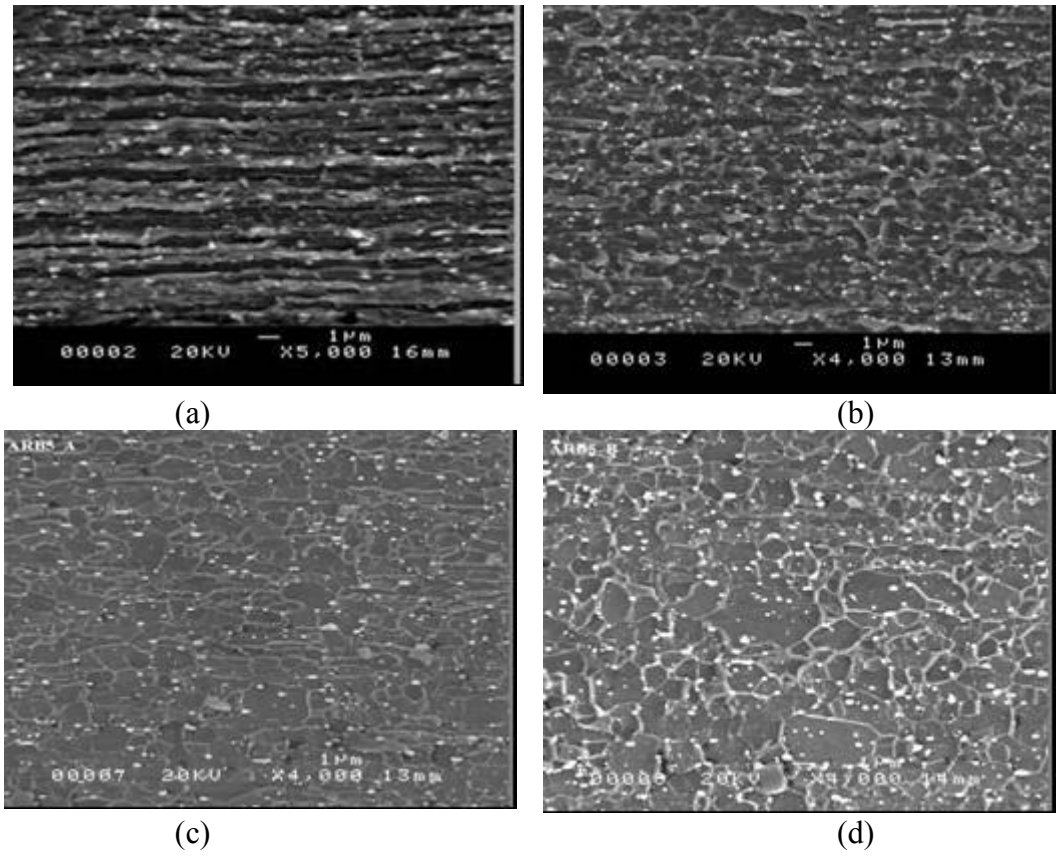


Fig. 4 – Schematic drawing of the Accumulative Roll Bonding Process.

In order to check the best conditions in terms of roll bonding temperature and soaking time to have deformation accumulation without excessive recovery or recrystallization, some tests were made at different temperatures in the range 600-700 °C and for different soaking times (1-15 min). The heating temperature before rolling pass, defined as the best compromise among no recrystallization, deformation accumulation, good bonding and decrease of rolling force was found to be 615 °C.
Therefore, before each rolling pass the sample was heated to 615 °C and maintained at this temperature for 2 min in order to homogenise the temperature of the sample. Using this rolling condition, it was noted that the minimum reduction needed for bonding was 40%.
The process was repeated 5 times for a total strain of about 4 (the total equivalent strain e after n cycles of the 50% roll-bonding ARB is $\sim 0.8\,n$).
Further it is important to notice that the layers included in a sheet treated after n ARB cycles at 50% deformation becomes 2^n. In this case, after 5 passes there will be 32 layers having each one about 120 μm thickness.

Results And Discussion

Microstructure.
Microstructural observations of as-ARB processed materials along the rolling direction (RD) showed more and more deformed structures going on the rolling sequence.
After the first pass, elongated grains due to deformation are observed and after the second pass a well deformed structure was detected. Increasing the total reduction, the structure is heavily deformed and it is no more possible to distinguish each layer by optical microscopy. The specimen severely deformed up to total strain of 4.0 exhibited the pancake shaped ultrafine grains, elongated along the rolling direction, as shown in Fig.5a, which were typically observed in the ARB processed materials [2-4]. A subsequent annealing procedure was carried out.
Samples after the 5th pass were annealed at 3 temperatures: 600, 650 and 700 °C for 3 minutes. In Fig. 5b the microstructure of the sample annealed at 600°C for 3 minutes is shown. Microstructure was quite homogeneous and polygonal grains were observed. Mean grain size was 0.8 μm.



(a) (b)

(c) (d)

Fig. 5 – Microstructures of ARB samples (a) as rolled after 5th pass, (b) annealed at 600 °C x 3 min (c) annealed at 650 °C x 3 min, (d) annealed at 700 °C x 3 min.

The microstructure of the sample annealed at 650 °C x 3 minutes (Fig. 5c) was quite homogeneous and polygonal grains were observed. In this case mean grain size was 1.7 μm, while in Fig. 5d the microstructure of the sample annealed at 700 °C x 3 min showed an inhomogeneous microstructure with some coarse grains, indicating that abnormal grain growth occurred. For this annealing condition the average grain size was 6 μm.

Mechanical properties

Tensile tests showed that decreasing the annealing temperature of ARB specimens (ε_{tot}=4) from 700 to 600 °C for 3 minutes, allows to increase the YS from 315 MPa (average grain size 6 μm) to 600 MPa (average grain size 0.8 μm), without significant effects on ductility (A_{50}%=14-15%). However, the yield to tensile ratio increased from 0.73 to 0.96. The material annealed at 650°C (average grain size 1.7 μm) exhibited an intermediate behaviour with YS=510 MPa and YS/TS =0.92.

Textures

In Fig. 6(a) the unique grain colour image of the sample annealed at 650 °C x 3 min is shown. Border lines between different colours represent high angle boundary grains with a misorientation angle exceeding 15°, whereas the fine lines inside the same colour area delineate low angle grain boundaries. This image shows that high angle boundary grains are not polygonal but elongated along the rolling direction. Inside the elongated grains there are a lot of small grains with low boundary angles. This is a typical situation of a recovered (and not recrystallised) material. It is confirmed in Fig. 6(b) where grain boundaries misorientation distribution shows a high fraction of low angles boundaries (53% against the 47% of high boundary angle).

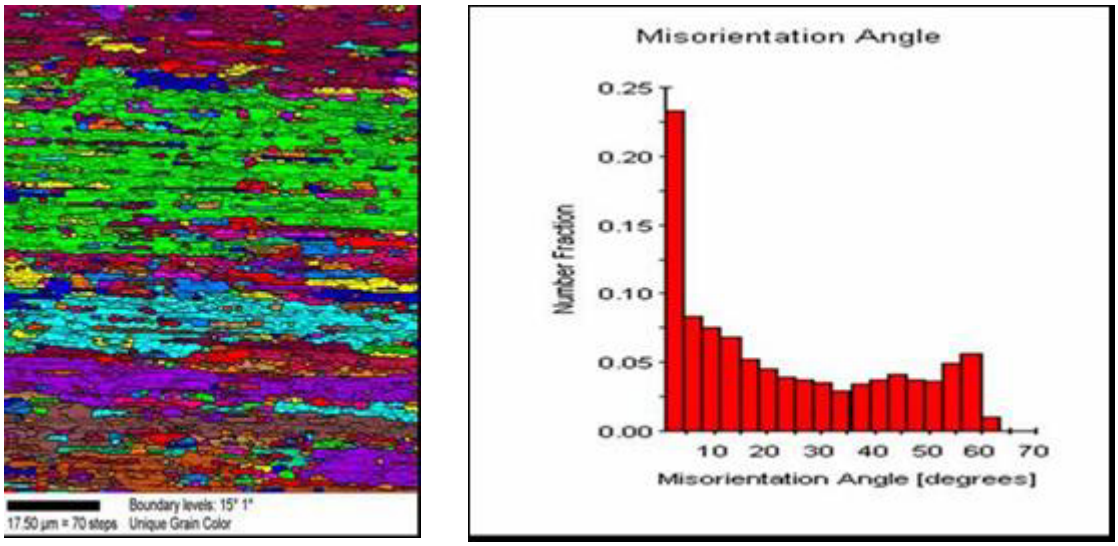


Fig. 6 – (a) Unique grain colour image of the sample annealed at 650 °C x 3 min, (b) Misorientation angle distribution.

SUMMARY AND CONCLUSIONS

It has been shown in this work that the ferrite grain size can be refined to 1-3 µm by different methods. They can be defined as advanced thermomechanical processes that can be easily applied on already existent pilot mills, and others that can be defined as severe plastic deformation techniques, like ARB, able to give large deformation presently not possible by conventional methods.
Different parameters were considered in designing a processing route to obtain fine grains and simultaneously suitable mechanical properties.
Mainly the carbon content and presence of microalloying elements influenced the best processing route to be applied to the chosen material.

By the DIFT mechanism, the level of ferrite refinement increased slightly when the carbon content increased, but in this case the critical strain for DIFT occurrence increased. Critical strain becomes higher and higher increasing the C content until it is quite difficult to activate this mechanism.
For DIFT to occur, the following hot rolling parameters should be controlled:

- Austenitization at low temperature in order to keep a small prior austenite grain size;
- Rolling reduction in the deformation temperature range Ar_3-Ar_3+30°C
- Minimum required strain is necessary, depending on carbon and Nb content.

As expected, large enhancements in strength and a reduced work hardening were observed in the ultrafine grained steels. This is reflected in the yield ratio (lower yield stress/ultimate tensile stress), which is around 0.9, compared with 0.7 for the conventional steels.

Accumulative Roll bonding showed to be very effective to refine grains imparting high strain.
This method, if extended to industrial scale, could give the possibility to obtain customized microstructures with improved combination of strength and ductility/toughness through adequate grain refinement (1-3 µm).

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