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# Development and Characterization of Steel Containing Very Fine Ferrite Grains

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**Ferrite grain sizes of the order of 1 to 2 μm were obtained by optimizing the strain, strain rate, the stage of cooling, as well as the cooling rate during hot rolling of 0.15C-0.92Mn-0.01Si-0.036S-0.04P-0.013Nb steel. It was found that in single-pass rolling of a 10 mm plate to a thickness of 3.5 mm with an entry temperature of 800 °C, and early-stage water cooling, very fine grains of ferrite (1-2 μm) were formed at the surface and in subsurface regions. It was also found that the threshold level of reduction during rolling, which is required for the refinement of ferrite grains, is >50%. The 3.5 mm thermomechanically processed plate was found to possess very attractive mechanical properties in terms of the yield strength (485 MPa), the ultimate tensile strength (UTS) (763 MPa), and particularly the yield strength to ultimate tensile strength (YS/UTS) ratio (0.63). This combination of properties can be explained on the basis of the composite microstructure consisting of ferrite and bainite that was obtained as a result of the thermomechanical processing.**

**Keywords** grain refinement, single-pass rolling, thermomechanical processing

## 1. Introduction

Grain size refinement in steels holds a great deal of promise due to the improvement in strength as well as toughness. Currently, ferrite grain refinement is achieved by a combination of controlled rolling and accelerated cooling. It has been found that this process can achieve a refinement of about 50% in steel plates of simple compositions. In the plate rolling of plain carbon-manganese (C-Mn) steel, grain size can be refined from 10 μm to ~5 μm when the plate is controlled rolled and accelerated cooled. This refinement in grain size raises the yield strength of the steel by about 80 MPa according to the well-known Hall-Petch relationship (Ref 1).

A further fivefold reduction in grain size to about 1 μm is expected to increase the yield strength by another 260 MPa. Another fivefold decrease in grain size to submicron ranges would raise the strength by a further 586 MPa to levels >1000 MPa.

In view of the enormous advantages that are to be gained by the refinement of the grain size for simple steel compositions, researchers are active all over the world trying to produce ultrafine-grained ferritic steels.

## 2. Current Status of Understanding

Presented below is a brief review (Ref 2, 3) of the current status of understanding of this research and of the developments that have taken place in recent years regarding attempts to produce ultrafine-grained ferritic steel. The following mechanisms have been proposed for obtaining ferrite grains as fine as 1 to 2 μm. These mechanisms are thought to operate during the thermomechanical processing of plain carbon steels and low carbon microalloyed steels.

Austenite is dynamically recrystallized, and the majority of deformation is obtained just above the austenite to ferrite transformation ($Ar_3$) temperature. This leads to the formation of ferrite grains that are 1 to 2 μm in size.

High-strain-rate deformation is carried out just below the $\gamma \rightarrow \alpha$ transformation temperature. Due to the heat generated as a result of the high-strain-rate deformation, the ferrite transforms to austenite for a while before transforming back to ferrite. This process, when carried out in a cyclic manner, leads to the formation of very fine ferrite grains.

The severe straining of ferrite is used to initiate dynamic recovery. However, this approach cannot lead to ultrafine grain sizes, but ferrite grain sizes of ~3 μm can be achieved. This occurs because further grain refinement in ferrite is very difficult due to the low strain-hardening exponent and the higher stacking fault energy of ferrite.

The deformation of coarse-grained austenite beyond a critical strain leads to the intragranular nucleation of ferrite inside the austenite grains, leading to considerable refinement of the ferrite grains. This mechanism is believed to operate while producing a layer of ultrafine ferrite at the surface of a thin strip. This refinement procedure requires austenitization at a high temperature to produce coarse austenite grains. The strip is then cooled to very near the $Ar_3$ temperature of the steel and is rolled at that temperature. An extremely high nucleation density of ferrite on the dislocation substructure within the coarse austenite grains is obtained due to the intense localization of shear strain in the layers close to the surface. This processing sequence results in a layer of ferrite approximately 1 μm in diameter close to the surface, while the core of the strip transforms to a more normal microstructure of coarser ferrite or bainite, depending on the cooling rate and composition of the steel.

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The unique features of this method of production of ultrafine-grained steel are the working characteristics that are not generally considered to be desirable in the conventional controlled rolling of steel, namely, a large austenite grain size, supercooling of the austenite, and high friction at the workpiece/roll interface.

Water quenching of the surface layer of the steel before the penultimate hot-rolling pass is another way to transform the surface layer of a strip into very fine ferrite grains. Heat from the core of the plate raises the surface temperature, so that the ferrite recrystallizes during the final pass, leading to a grain size of about 2 μm. This mechanism of refining the grain size at the surface is used when producing HiArrest plates (Nippon Steel, Kawasaki, Japan), which have superior crack-arresting properties.

An altogether different route of obtaining ultrafine grains is the cold rolling and warm annealing of a martensitic structure. However, the limitation of this processing route is the achievement of the martensitic structure in plain C-Mn steel, which limits the thickness of the sheet.

In the current study, a 0.013% Nb, C-Mn steel was thermomechanically processed in an attempt to obtain very fine grains of ferrite. The resulting microstructure was characterized.

## 3. Material

A 0.013% Nb, C-Mn steel was made in an air-induction furnace. The molten steel was poured into a mold to produce an ingot measuring 100 × 100 mm at the base. The ingot was hot rolled after soaking at 1200 °C in seven passes to a plate with final thickness of 10 mm, as per the following schedule:

$$100 \times 100 \text{ mm ingot} \rightarrow 85 \rightarrow 62 \rightarrow 45 \rightarrow 32 \rightarrow 22 \rightarrow 14 \rightarrow 10 \text{ mm}$$

The finish rolling temperature was about 850 °C. The plate was cooled in air. The composition of the steel was determined by optical emission spectroscopy, and the results are shown in Table 1. The average grain size in the 10 mm hot-rolled plate was found to be about ~20 μm.

## 4. Experimental

### 4.1 Dilatometry

A cylindrical sample having a 6 mm diameter and 90 mm length was machined from the 10 mm thick hot-rolled plate. The sample was subjected to dilatometry in a Gleeble 3500C (DSI, Chicago, IL) thermomechanical simulator. The $Ar_3$ temperature was determined at a cooling rate of 1 °C/s, which corresponds approximately to air cooling.

### 4.2 Thermomechanical Processing

The 10 mm hot-rolled plates were then thermomechanically processed in the following manner. The plates were soaked at 1250 °C for 30 min, then air cooled to about 820 °C (i.e., ~20 °C above the $Ar_3$ temperature). The plates were rolled at a roll peripheral speed of 4 m/min, and were reduced by 50%, 60%, and 65%. They were immediately cooled with water.

### 4.3 Optical Microscopy

Samples were prepared for optical microscopy from the starting hot-rolled plate as well as from the thermomechanically processed plates. The samples were machined from the middle portions of the plates to avoid end effects. The samples were polished and etched with 2% nital and were observed under a Neophot 30 (Germany) optical microscope. Grain sizes were determined using the in-lens micrometer in the eyepiece.

### 4.4 Microhardness Test

Vickers microhardness measurements were carried out using a 50 g load in the Leitz (Germany) microhardness tester. The load was applied to the sample for 10 s. Microhardness measurements were obtained in the region containing the fine grains of ferrite near the surface as well as in the interior bainitic regions of the sample.

### 4.5 Determination of the Prior Austenite Grain Size

To determine the prior austenite grain size, a cylindrical sample having an 8 mm diameter and a 12 mm length was machined from the 10 mm thick hot-rolled plate. This sample was heated to 1250 °C and held for 5 min. It was then cooled at a rate of 1 °C/s to a temperature of 800 °C followed by water quenching in the Gleeble 3500C thermomechanical simulator. Metallographic samples were prepared from the thermomechanically simulated specimen. The samples were polished and etched with 2% nital. Although nital is not the proper etchant for delineating prior austenite grain boundaries, the size of the prior austenite grains in this sample could still be delineated.

### 4.6 Tensile Testing

Tensile samples having a gage length of 25 mm were machined from the thermomechanically processed 3.5 mm thick plate. These samples were tensile tested at a crosshead speed of 5 mm/min in an Instron 1195 (UK) tensile testing machine.

**Table 1 Chemical composition (optical emission spectroscopy)**

| Composition, wt.% | | | | | |
|---|---|---|---|---|---|
| **C** | **Mn** | **Si** | **S** | **P** | **Nb** |
| 0.15 | 0.92 | 0.01 | 0.036 | 0.039 | 0.013 |

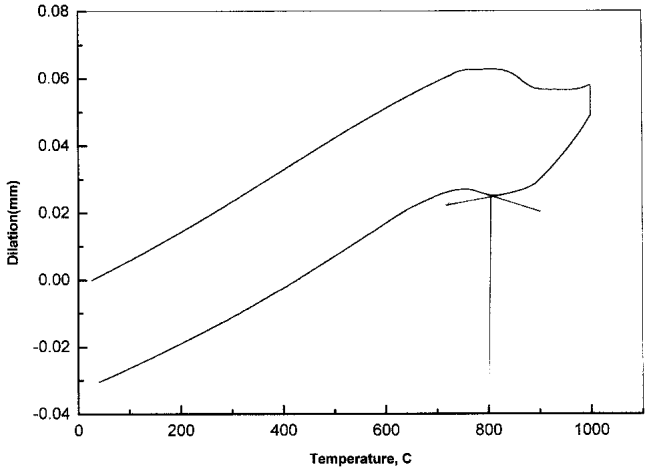



**Fig. 1** Dilatometric measurement showing $Ar_3$ temperature

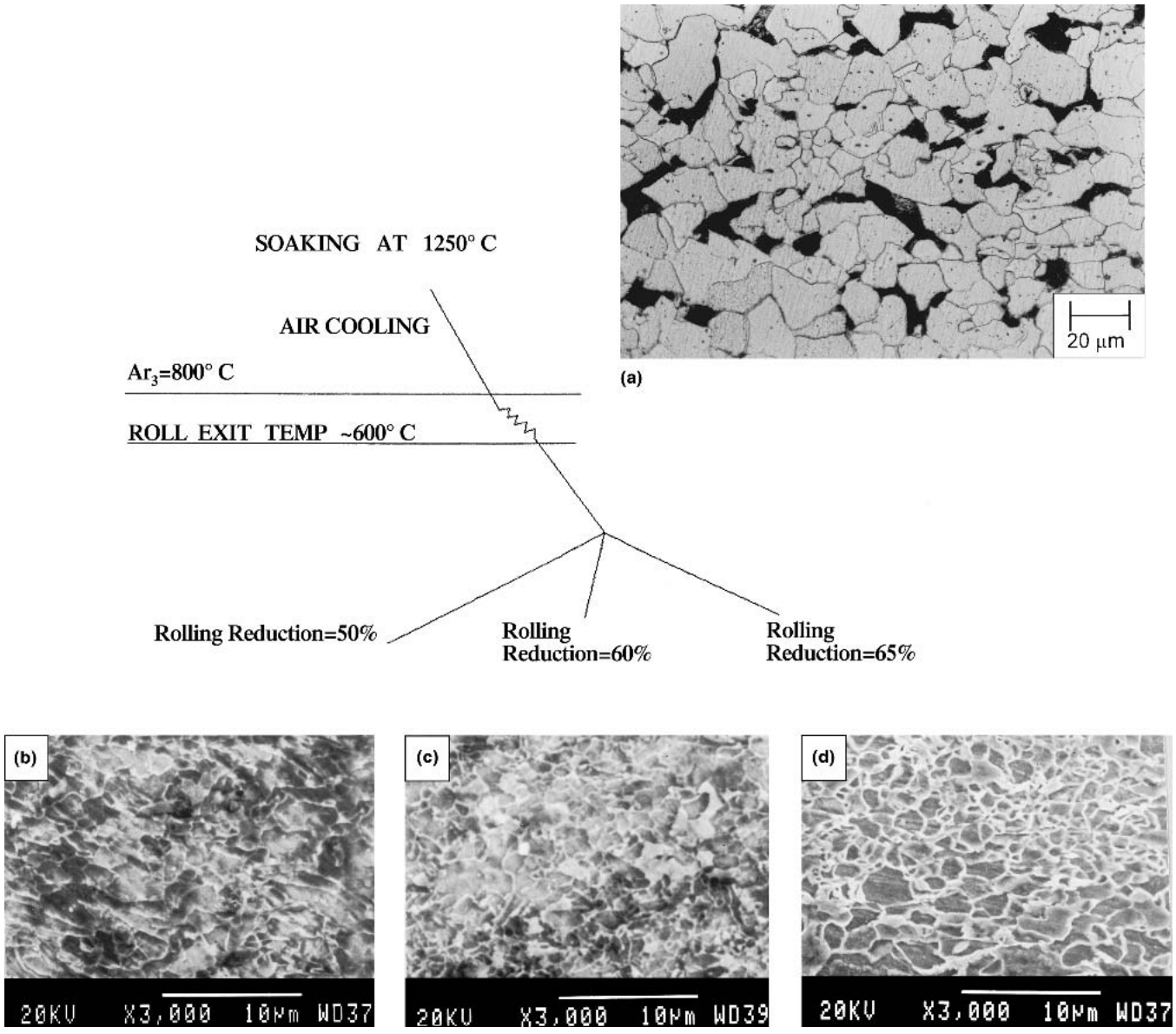



**Fig. 2** Schematic depiction of the thermomechanical processing. (a) Optical micrograph of the starting material showing average ferrite grain size (~20 μm). (b) SEM micrograph of the region near the surface showing average ferrite grain size (3.5-4.5 μm. (c) SEM micrograph of the region near the surface showing average ferrite grain size (1-2 μm). (d) SEM micrograph of the region near the surface showing average ferrite grain size (0.8-2 μm)

### *4.7 Scanning Electron Microscopy*

The polished and etched samples from the 3.5 mm thick thermomechanically processed plate as well as the fracture sur face of the sample broken in the tensile testing program were observed in the JSM 840A (Japan) scanning electron microscope (SEM).

## 5. Results and Discussion

The 10 mm hot-rolled plates were soaked at 1250 °C for 30 min. They were taken out of the furnace and cooled in air down to a temperature of ~820 °C, which is ~20 °C above the $Ar_3$ temperature (Fig. 1). Reductions of 50%, 60%, and 65% were imposed on the plate in single passes at "a." The plates were immediately cooled with water. Figure 2 is the schematic depiction of the thermomechanical processing sequence that was adopted in the current study. The slope shown in the portion indicating deformation in the cooling curve is attributed to any cooling that might occur during rolling. The optical micrograph of the 10 mm thick hot-rolled plate is shown in Fig. 2(a). The initial microstructure consists of ferrite and pearlite, with an average grain size for ferrite of 15 μm. Upon further thermomechanical processing, it was observed that there had been little grain refinement with the 50% reduction. However, considerable grain refinement occurred when the rolling reductions were 60 and 65%. Figure 2(b) to (d) show the SEM micrographs of the surface regions obtained from the samples that had been given reductions of 50, 60, and 65%, respectively, during their thermomechanical processing sequence. In view of the pronounced degree of grain refinement that was observed in the sample, wherein the reduction involved during thermomechanical processing was 65%, further characterization of the plate was carried out.

Because the plates were austenitized at 1250 °C, there was considerable coarsening of the austenite grains (Fig. 3). Air

cooling of the plates down to just above the $Ar_3$ temperature (~800 °C) did not affect the grain size. Figure 4(a) shows the through-thickness SEM micrograph of the sample. Figure 4(b) shows the SEM micrograph of the region from the interior of the sample. Figure 4(c) shows the optical micrograph of the surface region. It was observed from these micrographs that the region near the surface consisted of extremely fine ferrite grains. The interior of the sample, as is evident from the micrograph, consisted of bainite. Therefore, the 3.5 mm thick thermomechanically processed plate consisted of a composite microstructure of bainite sandwiched between two layers of extremely fine ferrite grains. The deformation of coarse austenite grains just above the $Ar_3$ temperature is known to induce intragranular nucleation of ferrite in the austenite grains. It is also known that a minimum delay between deformation and transformation leads to more numerous ferrite nucleation sites as a result of deformed and unrecovered austenite grains. In the present case, all of the samples were cooled immediately after rolling. Although it is believed (Ref 4) that simultaneous deformation and transformation is most desirable for the ultrarefinement of grains, in the present case the delay between deformation and cooling was very small. The sample exhibited extremely fine grains of ferrite at the surface to a depth of approximately 0.5 mm.

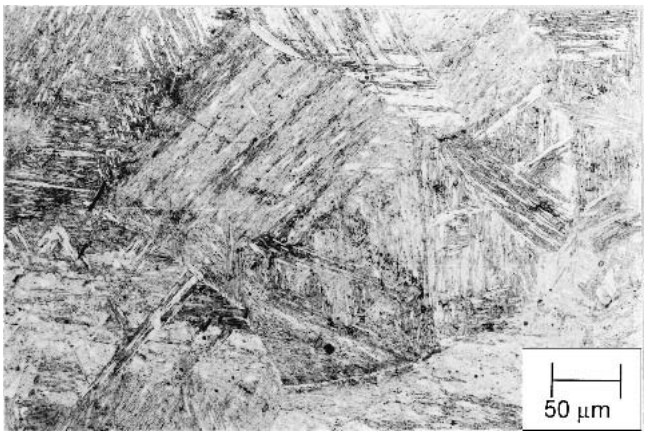



**Fig. 3** Optical micrograph showing prior austenite grains

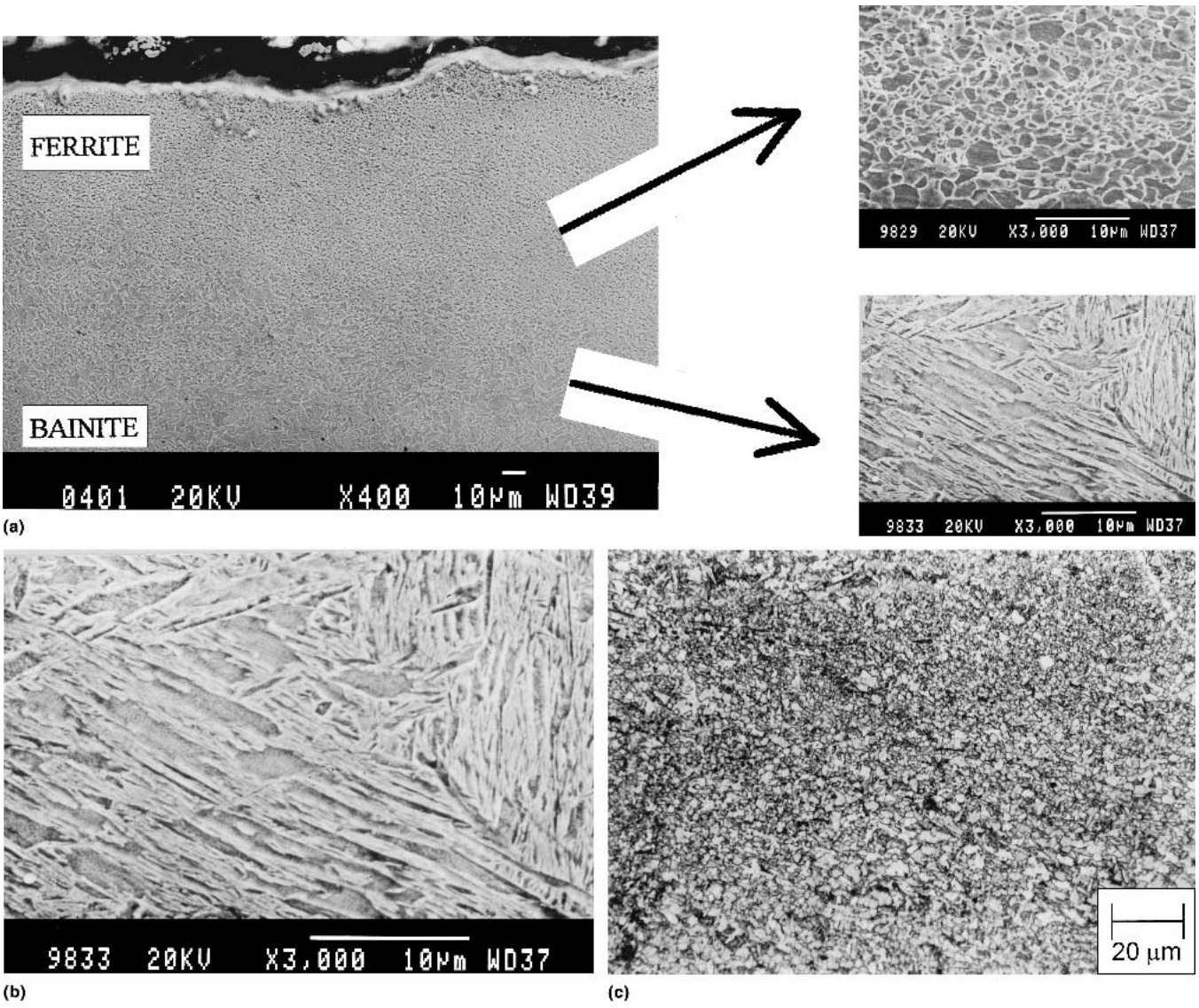



**Fig. 4** (a) SEM micrograph showing composite microstructure. (b) SEM micrograph showing bainitic microstructure. (c) Optical micrograph showing ultrafine grains of ferrite

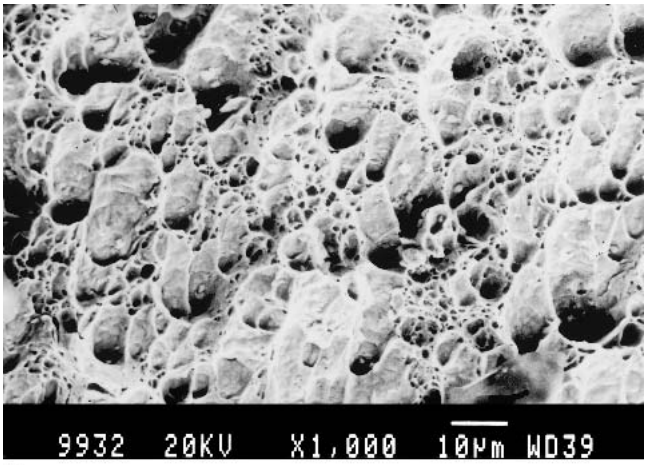
Table 2 shows the results of the microhardness tests carried out on the sample. The region near the surface, consisting of fine ferrite grains, had a microhardness of 349 HV, whereas the interior possessed a hardness of 453 HV. Table 3 displays the results from the tensile tests. A yield strength (YS) of 485 MPa, an ultimate tensile strength (UTS) of 763 MPa, and an elongation of 12% were obtained. These tensile properties are quite attractive, especially in view of the low YS-to-UTS ratio of 0.63 that has been obtained at these levels of YS and UTS (Ref 5). Normally, an increase in the UTS is accompanied by an increase in the YS, leading to a higher YS-to-UTS ratio (Ref 6). However, in the present case, the YS/UTS ratio that has been obtained is relatively low, particularly at the level of YS that has been achieved. The ratio of YS to UTS is quite favorable, especially for forming applications. This has been achieved due to the composite microstructure.

**Table 2 Microhardness test results**

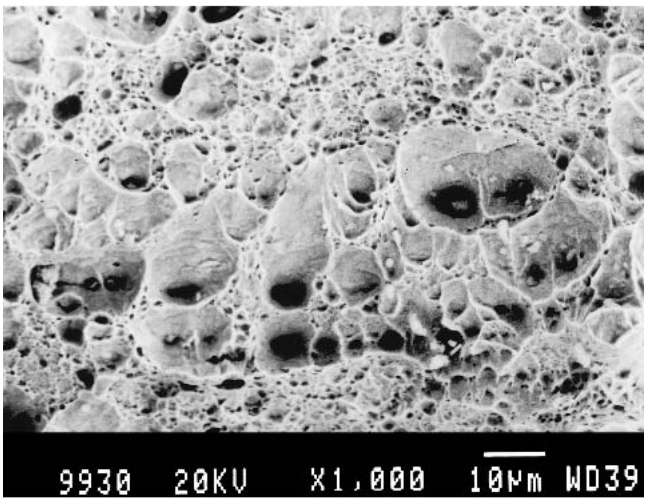
| Phase | Vickers hardness, HV |
|---|---|
| Ferrite | 349 |
| Bainite | 453 |

**Table 3 Average tensile properties**

| YS, MPa | UTS, MPa | Elongation, % | YS/UTS |
|---|---|---|---|
| 485.0 | 763.0 | 12.0 | 0.63 |




(a)




(b)

**Fig. 5** (a) SEM micrograph of the fracture surface near the edge. (b) SEM micrograph of the fracture surface of the interior

Shown in Fig. 5 are the SEM micrographs of the fracture surfaces of a tensile sample that was broken during tensile testing. Figure 5(a) shows the fractograph from the surface region, whereas the fracture surface from the interior of the sample is shown in Fig. 5(b). These fractographs predominantly show dimpled features, which are indicative of ductile fracture. These observations reinforce the fact that this kind of composite microstructure gives rise to attractive tensile properties. It is known that lower bainite possesses superior toughness properties due to the fine carbide distribution, whereas extremely fine ferrite grains confer excellent crack-arresting characteristics. Hence, the composite structure of these two phases has resulted in a material with superior tensile properties.

## 6. Conclusions

The following conclusions can be drawn from the current study:

- Thermomechanical processing of 0.15C-0.92Mn-0.01Si-0.036S-0.04P-0.013Nb steel, which involved soaking at 1250 °C, roll entry at 800 °C, a rolling reduction of 65%, and early-stage water cooling, resulted in the formation of a composite microstructure consisting of bainite sandwiched between layers of extremely fine-grained ferrite (1-2 μm in size).
- The threshold rolling reduction for the formation of such a microstructure is about 50%.
- A plate processed in such a manner exhibited very attractive tensile properties, particularly in terms of YS-to-UTS ratio.

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