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# Ultrahigh strength and low yield ratio of niobium-microalloyed 900 MPa pipeline steel with nano/ultrafine bainitic lath

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#### 1. Introduction

There is a continued and increasing demand to transport oil and gas by pipelines at high operating pressures in the attempt to increase the capacity. This requires the use of ultrahigh strength steels. Furthermore, increasing the strength of the pipeline steel allows wall thickness to be significantly reduced with consequent reduction in weight. Thus, a major goal within the steel industry is to develop ultrahigh strength microalloyed pipeline steels with high toughness and formability. High toughness is important to reduce stress-induced cracking, while formability is important from the view point of pipe-bowing. Thus, high strength in conjunction with high toughness and formability are important requirements of the pipeline industry for transporting natural gas and crude oil over a long distance at high pressures [1–3]. Other characteristics that are required include resistance to hydrogeninduced blister cracking in sour service environment [4,5], stress corrosion cracking resistance, especially in H<sub>2</sub>S environment [6–8], and fatigue resistance [9,10]. In the aforementioned regard, it is important that the microstructure of the pipeline steel provides a combination of aforementioned properties.

#### ABSTRACT

An ultra-low carbon niobium-microalloyed steel with yield strength of ~900 MPa has been processed on a pilot-plant scale. The microstructure of the steel is primarily characterized by lower bainite and acicular ferrite, with small fraction of lath-martensite and martensite–austenite (MA) constituents. Bainite is present as fine domains. A combination of niobium and titanium precipitates was observed at the grain boundaries and in the interior of the grains and includes irregular (~40–150 nm of (Nb, Ti)(C, N)) and fine cuboidal/spherical particles of NbC (~30–50 nm). It was observed that accelerated cooling inhibited the precipitation of Nb and Ti carbides. The Charpy impact toughness at -20 °C was 200 J and tensile elongation was 15% with the yield ratio of less than 0.84. The good matching of high strength and low yield ratio was realized by two-stage thermo-mechanical rolling combined with fast cooling.

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It is known that the microstructure is dictated by alloy chemistry and thermo-mechanical processing. The alloying elements commonly added in pipeline steels to obtain the desired microstructure and mechanical properties [5,6,11–13] include Mn, Nb, V, Ti, Mo, Ni, Cr and Cu. However, judicious selection of alloying elements is necessary to obtain beneficial effect on mechanical properties with reduced alloy cost. For instance, the number and amount of alloying elements are reduced to achieve lower carbon equivalent (CE) to ensure good field weldability [2,3]. On the other hand, alloy additions such as Cr, Cu, and Ni are added to obtain strength in severe corrosive environment [2,5].

In the processing of pipeline steels, **c**ontrolled thermomechanical processing is the preferred route for the development of API grade pipeline steels because it provides the desirable and fine-grained microstructure. Furthermore, it allows high strength-toughness combination to be obtained via accelerated cooling [3], where the final microstructure depends on processing parameters including reheating temperature, percentage reduction, deformation temperature, cooling rate, and cooling temperature [14]. This is because fine austenite grains, substructure and dislocations in austenite effectively promote the transformation of fine ferrite. The primary grain refinement in controlled rolling is achieved through recrystallization of austenite during deformation and through the use of microalloying elements, such as Nb that precipitates as fine carbides and inhibits grain growth [14].

During thermo-mechanical processing of pipeline steels [1,7–9,11,15–20], different combinations of microstructures are obtained. Lower bainite and acicular ferrite microstructure with

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Table 1	l
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Chemical composition of ultrahigh strength niobium-microalloyed pipeline steel.

С	Si	Mn	Р	S	Cu	Ni + Mo	Nb	V	Ti
0.01-0.06	0.20-0.40	1.50-2.10	<0.008	<0.002	0.10-0.30	0.20-0.70	0.060-0.100	0.030-0.060	0.010-0.020

Tal	ble	2

Mechanical properties of ultrahigh strength niobium-microalloyed pipeline steels (plate thickness 12.5 mm).

Heat no.	Direction	Yield strength (MPa)	Tensile strength (MPa)	Yield ratio	Impact toughness at $-20^{\circ}C(J)$
WH090302	Т	860	1020	0.84	217, 218, 230 (avg. 222)
WH090302	L	850	1010	0.84	230, 222, 226 (avg. 226)
WH090303	Т	925	1110	0.83	227, 208, 230 (avg. 222)
WH090303	L	910	1090	0.83	245, 244, 235 (avg. 241)
WH090306	Т	905	1055	0.85	201, 207, 234 (avg. 214)
WH090306	L	890	1055	0.84	226, 202, 203 (avg. 210)

uniform distribution of martensite/austenite (M/A) islands as a second phase, is considered to provide the desired mechanical properties [1,11,14,21]. Based on the above background, an ultrahigh strength 900 MPa Nb-microalloyed pipeline steel with a low yield ratio of 0.84 has been processed on a pilot-plant scale. We describe here the structure and mechanical properties of ultrahigh strength steel with high toughness.

#### 2. Experimental

The pipeline steel was electric arc furnace melted, hot-rolled into (12.5 mm) plate and cooled to a lower temperature by accelerated cooling device. The chemical composition of the steel is presented in Table 1. The thermo-mechanical processing details are not described here in detail due to proprietary reason. The percentage of reduction in the non-recrystallization zone was 35-52% and in the recrystallization zone was 60-70%. The cooling rate was 10-35 °C/s with coiling temperature of 250-450 °C.

Specimens were cut from the plate and mounted for metallographic examination. Standard grinding and polishing techniques were employed and specimens etched with 2% nital. Optical microscopy (OM) and scanning electron microscopy (SEM) imaging techniques were used to examine the microstructure at low magnification. The electron back-scattered diffraction (EBSD) was employed to measure the domain size of the microstructure by setting the misorientation angle of adjacent boundary as 10°. The metallographic measurements of the domain size were made on at least 10 fields of view in order to obtain representative data for EBSD analysis on Quanta 400 SEM.

Transmission electron microscopy (TEM) was carried out on thin foils prepared by cutting thin wafers from the small coupons and grinding them to  $60-70 \,\mu$ m in thickness. Three millimeter discs were punched from the wafers and electropolished using an electrolyte solution of 10% perchloric acid. These foils were examined by JEM2100F TEM operated at 200 kV and Hitachi 7600 TEM operated at 120 kV. The carbon extraction replica technique was used to examine precipitates. To prepare carbon extraction replicas, the polished specimens were etched with 2% nital, followed by evaporation of carbon evaporated onto the etched surface. Finally, the surface was scored to  $\sim$ 3 mm squares and the sample etched first with 10% nital and then with 2% nital. Subsequently, the extracted replicas were rinsed with distilled water and placed on the copper grid and dried.

Standard tensile tests were conducted at room temperature on transverse and longitudinal specimens machined according to ASTM E8 specification. Impact toughness was measured using Charpy V-notch impact test according to ASTM 23 at -20 °C.

#### 3. Results

# 3.1. Mechanical properties of ultrahigh strength niobium-microalloyed pipeline steel

The yield strength, tensile strength, elongation and Charpy toughness for ultrahigh strength niobium-microalloyed pipeline steel with ultra-low carbon are summarized in Table 2. The yield strength and tensile strength are in the narrow range of 850-925 MPa (121-132 ksi) and 1010-1110 MPa (144-158 ksi), respectively. The Charpy V-notch impact toughness at -20 °C was in the range of 148-170 Nm (201-230 J). The yield ratio was in the range of 0.83-0.85, and is lower than currently available high strength pipeline steels. The mechanical properties were similar in longitudinal and transverse direction.

#### 3.2. Microstructure of the ultrahigh strength pipeline steel

In Fig. 1 the light and scanning electron micrographs are presented. The microstructure predominantly consists of lath-bainite



Fig. 1. (a) Light and (b) scanning electron micrograph of 900 MPa (120×) pipeline steel showing primarily lath-type bainite and small fraction of acicular ferrite.

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