

Contents lists available at ScienceDirect

Materials Science & Engineering A



journal homepage: www.elsevier.com/locate/msea

Microstructure and mechanical property of in-situ nano-particle strengthened ferritic steel by novel internal oxidation



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ARTICLE INFO

Article history: Received 4 April 2014 Received in revised form 6 May 2014 Accepted 9 May 2014 Available online 16 May 2014

Keywords: in situ nano-particles Titanium oxide In melt Precipitation hardening

ABSTRACT

A novel route of fabricating nano-particles strengthened ferrite steel was investigated in this study. Rather than by externally adding nano-oxide powders, we adopted the endogenous method of controlling oxide reaction and solute concentration distribution in the process of deoxidization to obtain a high density of in-situ nano-oxide particles homogeneously dispersed in the ferrite matrix in melt. The microstructure and tensile properties of these materials had been investigated to clarify the interrelation between the composition, microstructure and mechanical properties. Transmission electron microscopy (TEM) analysis indicated that these nano-particles were titanium oxides, which have a positive effect on optimizing inclusions and refining grains. Tensile tests revealed that these titanium oxide particles play an important role in increasing the yield strength. The steel has yield strength of 711 MPa, approximately three times higher than that of conventional plain carbon structural steels, and its ultimate tensile strength reaches 810 MPa with an elongation-to-failure value of 22%. Precipitation hardening and grain refinement hardening are the dominant factors responsible for yield strength increasing in this steel.

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1. Introduction

At present, nano-particles strengthened ferritic steels are typically referred to as oxide dispersion strengthened (ODS) steels by externally adding nano yttrium oxide powders [1]. As summarized by Schneibel et al. [2], the manufacturing of ODS ferritic alloys usually takes a long time (e.g., one day) in a high-energy ball mill, followed by hot consolidation. The mechanical alloying process for creating oxide dispersoid is expensive and energy-intensive. Also, mechanically alloyed materials typically develop pores during high temperature annealing [3]. These pores degrade the mechanical properties. Issues for ODS ferritic or ferritic/martensitic steels processed by mechanical alloying (MA) are anisotropic mechanical properties due to the bamboo-like structure and impurity pick up during MA [4]. Long time high-energy mechanical milling could bring contamination from the atmosphere and the milling media; the effects of such contamination on structure, mechanical properties, and irradiation performance of the MA products could be vital. Furthermore, the nano-particles strengthened steel prepared by adding nano oxide particles externally generates such problems like the uneven distribution or the press that is easily caused by

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http://dx.doi.org/10.1016/j.msea.2014.05.020 0921-5093/© 2014 Elsevier B.V. All rights reserved. the interface between the particle and the matrix. Compared to external adding, the endogenous method does not need to prepare nano powder. It is low-cost, time saving and has high efficiency. The in-situ nano-particles prepared in this way are more dispersive, uniform and present a coherent or semi-coherent relationship with the matrix. The formation of coherent or semi-coherent interface will reduce the interfacial energy between oxide particle and matrix, which results in the increasing stability of the oxide particles [5].

The objective of this work is to take advantage of the in-situ nano oxide particles produced by a novel internal oxidation to strengthen the ferritic steel. The manufacturing route, microstructure and mechanical properties of the steel have been preliminarily investigated.

2. Experimental methods

Two steels, with and without titanium, were prepared and studied. The ingots were produced by vacuum induction melting furnace and the main chemical compositions of the steel investigated are shown in Table 1. Steel B has the same composition as that of steel A except the addition of 0.15% Ti. The former adopted a special route to produce nano precipitation in melt directly. Here we show the nanoparticles formed in melt directly which is

Table 1

Main chemical composition of steels investigated (wt%).

Steel A	C 0.068	Si 0.13	Mn 0.28	S 0.030	P 0.024	N 0.004	-	0
Steel B	C 0.068	Si 0.13	Mn 0.28	S 0.030	P 0.024	N 0.004	Ti: 0.15	< 40 ppm



Fig. 1. Schematic of the processing method for the formation of nanoparticles in melt through a combination of multipoint dispersion supply processing and electromagnetic stirring.

achieved by solidification processing through a combination of uniform dispersion supply processing and electromagnetic stirring. Fig. 1 schematically shows the specific experimental process. First, the base metal is placed in the vacuum melting furnace. After it fully melts and the solidification temperature of the molten steel is in the range from 1530 °C to 1620 °C, the pure Ti wire is added in the way of multipoint dispersion supply. It is worth noting that electromagnetic stirring (\sim 4 KHz frequency) always exists during the whole melting process. Under the effect of electromagnetic stirring, flowing linear velocity of the molten metal is about 10–20 m/s. It is followed by thermal insulation for about 60 s after all the pure Ti wires completely react. In the casting process, the flowing linear velocity of the molten metal is not less than 1.7 m/s. Finally, the melt was cooled down inside the crucible in air over a cooling rate of about 500 °C/min in the process of solidification.

Subsequently, two ingots with dimensions of 135 mm \times 75 mm \times 96 mm were austenitized at 1200 °C for 2 h, then hot rolled into 11 mm thick plate by seven passes. The initial and final rolling temperatures were 1150 °C and 870 °C, respectively. Finally the specimens were water-cooled to a certain temperature (\sim 600 °C) and then air-cooled to ambient temperature.

Metallographic specimens were cut perpendicular to the roll direction from hot plate and were ground, polished, etched with 3% nitric acid–alcohol and then observed with 9XB-PC optical microscopy. Microstructure of the specimen was further investigated by using JEOL JEM-2100 TEM operated at 120 kV. TEM foil was prepared by cutting a thin wafer from the steel samples, followed by mechanically thinning them to ~35 µm in thickness. Three millimeter discs were punched from the wafers and electrochemically-polished using a solution of 10 vol% HClO₄– methanol electrolyte at low temperature. The foils were thinned further for electron transparent area using focused ion beam.

According to ASTM E8 specification, dog-bone shaped plate on longitudinal specimens with a gauge section of 30 mm Length \times 20 mm Width \times 2 mm Thickness were used for the tensile tests,

which were conducted at room temperature with a strain rate of 10^{-3} s⁻¹ using an FPZ 100 machine.

3. Results

3.1. Microstructure

Fig. 2a shows the morphology and distribution of the inclusions using TEM. Many cubic inclusions about several hundred nanometers can be observed in steel A. However, as Fig. 2b shows, a remarkable decrease in the inclusion number can be observed in steel B. The inclusion shape changes from cubic into circular and it locates in the intersections of grain boundary. It is easy to see the inclusion pinning on the grain boundary which will stop the grains from growing up.

Energy Disperse Spectroscopy (EDS) is used to determine the constitution of the inclusion in the two steels. Results indicate that the cubic inclusions are MnS while the circular one is a composite inclusion of MnS and Titanium Oxide.

Fig. 3 shows the optical micrographs of the investigated steels. It can be seen that the average grain size of steel B is obviously smaller than that of steel A, due to the comprehensive function of the nano precipitated phase.

3.2. Characterization of nano precipitates

Fig. 4 shows representative TEM micrographs in the investigated steel. Without adopting special craft, there are no nanoparticles in steel A, as shown in Fig. 4a. On the contrary, it is obvious to see that a large number of nano-particles are homogeneously distributed in the matrix with mean diameter of approximately 5 nm and sizes smaller than 10 nm in steel B, as shown in Fig. 4(b, c). Fig. 4d shows the corresponding darkfield image. Download English Version:

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