



Nb segregation at prior austenite grain boundaries and defects in high strength low alloy steel during cooling



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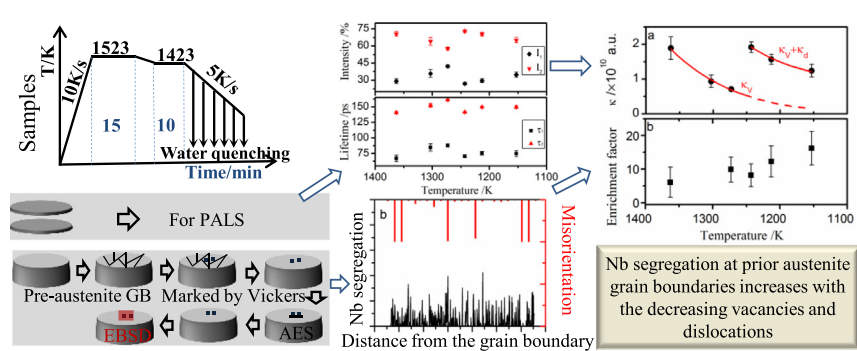
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HIGHLIGHTS

- Segregation of Nb at prior austenite grain boundary during cooling is characterized by Auger electron spectroscopy.
- Behavior of vacancy type defects during cooling are studied using positron annihilation lifetime spectroscopy.
- The segregation of Nb at prior austenite grain boundary increases with decreasing temperature.
- Vacancies and dislocations may both contribute to the segregation of Nb.

GRAPHICAL ABSTRACT



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ABSTRACT

The segregation of Nb at prior austenite grain boundary during cooling was investigated. Auger electron spectroscopy and electron backscattering diffraction were performed to characterize the segregation of Nb, while positron annihilation lifetime spectroscopy was applied to analyze the behavior of defects within the material. The formation energy of vacancy and the activation energy of the climb or glide of dislocations coincide well with reported results. The segregation of Nb at the grain boundaries increases with the decreasing defects during the cooling process. The diffusion of vacancy and the climb or glide of dislocations may both contribute to the segregation of Nb.

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1. Introduction

High-strength low-alloy steel (HSLA) has been widely used in modern technology due to its high strength and great weldability [1–6]. To improve the mechanical properties, different alloy elements have been added to the steel. Nb is one of the most frequently used elements. Nb

has great effect on the phase transformation behavior and the final microstructure because of its great interaction with interfaces. Quite a few studies on the mechanism of Nb have been reported [7] and two basic models have been suggested: (i) the grain boundary can be pinned by tiny Nb(C, N) precipitate [8–11]; and (ii) the growth of the austenite grain can be inhibited by solute Nb with a solute drag effect [12–15].

Nb precipitates have been sufficiently studied using TEM [16–18], whereas the researches on solute Nb are less sufficient because of the experiment difficulties. Solute Nb has great effect on the static recovery

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and recrystallization behavior, and this effect is especially important at high temperature before Nb precipitate occurs [7,19,20]. A few studies on solute Nb measured by a three-dimensional atom probe (3DAP) have been reported [21–23]. These studies mainly focus on the direct observation or quantitative measurements of Nb segregation at interfaces. As is already known, alloying elements and heat treatment are two crucial factors that affect the final mechanical properties of steel, and this draws our attention to the behavior of Nb during the cooling process.

The development of segregation is always associated with diffusion process which is very difficult to have a direct characterization, especially at high temperature. Fortunately, Nb, with a large atomic size, occupies a substitutional site in Fe matrix and diffuses with a vacancy-mediated mechanism [24]. Therefore, the diffusion of Nb is affected by the diffusion of vacancies and could be deduced from the diffusion of vacancy.

Positron annihilation lifetime spectroscopy (PALS) is powerful in characterizing the vacancy type defects in metals and alloys. By recording the time intervals between the emitting during the decay of ^{22}Na and annihilation with an electron in the material of the positrons, a lifetime spectrum is obtained. The lifetimes of positrons that annihilate with electrons at different regions are different, e.g. the positrons trapped by vacancy have a long lifetime, while the positrons that annihilated with a free electron have a short lifetime. Therefore, in principle, the type and concentration of defects can be resolved from the spectrum.

The characterization of distribution of Nb at grain boundary is easy to be impacted by signals from matrix if the technique has a large interaction volume, since it is difficult to make the grain boundary perpendicular to the scanning interface. Auger electron spectroscopy (AES) is used to characterize the distribution of Nb as it has an extreme sensitivity to surface species due to the short mean free path of Auger electron.

In this work, the behavior of Nb at grain boundaries during the cooling process is studied. The distribution of Nb at prior austenite grain boundaries (the austenite grain boundaries preserved by water quenching) within the quenched samples was characterized by AES and electron backscattering diffraction (EBSD), whereas the vacancy type defects was measured by PALS.

2. Materials and methods

2.1. Materials and the heat treatment

The material used in this work is a low-carbon microalloyed steel prepared by induction melting. The chemical composition of this material were as follows: (wt.%) 0.044C, 0.22 Si, 1.67 Mn, 0.0083 S, 0.0065 P, 0.046 Nb, 0.016 Ti, 0.002 B and 0.46 Cu. The ingot was soaked at 1473 K for 2 h and then forged to 14 mm bars. The bars were machined to φ 10 mm \times 30 mm dumbbell samples with an 8 mm \times 10 mm dumbbell bar. Heat treatment was conducted on a Gleeble 1500 thermal simulator. The specimens were first heated to 1523 K at a rate of 10 K/s and held for 15 min to ensure Nb is fully solute, then cooled down to 1423 K at a rate of 2 K/s and held for 10 min. Subsequently, the specimens were cooled down at a rate of 5 K/s to 1363 K, 1303 K, 1273 K, 1243 K, 1213 K, and 1153 K, respectively and quenched in water immediately to keep the microstructures.

2.2. AES and EBSD measurements

After the heat treatment, two sets of samples were cut off from the dumbbell bars. The first set of samples included the φ 8 mm \times 5 mm bars for AES and EBSD measurements, whereas the other set included couples of φ 8 mm \times 1 mm slices for PALS measurement. All samples were mechanically polished for subsequent measurements.

The difficulty of this experiment lies in the fact that AES and EBSD measurements must be conducted at the same prior austenite

boundaries. This challenge was overcome by marking the prior austenite boundaries with Vickers hardness indentation. Before the AES measurement, all samples were etched with a solution of 4% nitric acid and 96% alcohol to obtain 4% nital to reveal the grain boundaries. The long and straight prior austenite grain boundary was chosen and marked on each sample. The samples were mechanically polished again to eliminate etched layer and the markers should remain clear during and after polishing.

AES measurement was conducted on a PHI 700 AES system (ULVAC-PHI, Japan). The samples were first sputtered by Argon ion sputtering to get a clean surface. Then, AES line scanning was carefully performed perpendicular to the grain boundary with the aid of the markers. Auger electrons were excited by an electron beam of 5 keV. The beam size was estimated to be 30 nm and the distance between two adjacent scanning points was no longer than 150 nm. Every spectrum was accumulated 60 times to get a credible result.

After AES scanning, the specimens were electrochemically polished by a solution of 5% perchloric acid and 95% alcohol. EBSD measurement was conducted in an HKL NordlysMax EBSD system (Oxford Instruments) which was equipped on a scanning electron microscope (Carl Zeiss). The misorientation of the grain boundaries can be obtained from the EBSD results.

2.3. PALS measurements

PALS measurements was conducted at room temperature (293 K) using a slow-fast coincidence system. The time resolution was approximately 196.5 ps (FWHM). A ^{22}Na positron source was used and sandwiched between the couple of samples. The total coincidence events are accumulated to over 2×10^6 counts for each spectrum. Using the fitting software LT 9.0 [25], the spectrum was decomposed into two components after subtracting the source and background components, which means that the positrons have two lifetimes (τ_1 and τ_2). The proportion of the two components are I_1 and I_2 , respectively, and have the relation $I_1 + I_2 = 1$.

3. Results and discussion

3.1. Nb segregation

The microstructures obtained by water quenching are mainly lath martensitic and the average lath size is about 1.5 μm . The average prior austenite grain size was about 70 μm and the grain size becomes more uniform with the decrease of the quenching temperature.

The sensitivity of AES is tested before the experiments conducted. The sample quenched at 1243 K was first measured by the method introduced in this paper and the result is shown in Fig. 1(a). Then, the sample was etched with a solution of 4% nitric acid and 96% alcohol and measured by AES again (with the same measure parameters). The result is shown in Fig. 1(b).

Fig. 1 shows that the solute Nb may dissolve out of the sample when etched by the solution, and the intensity in Fig. 1-b is obviously lower than that in Fig. 1-a. Besides, Fig. 1-b shows that there are few noises in the AES spectrum, and most of the peaks are associated with Nb atom in sample.

To compare the segregation of Nb in different samples, the results of AES are normalized as follow. First, all the peak values are averaged and the average value is proportional to the concentration of Nb in material. Then, the normalized distribution of Nb is obtained by dividing all the peak values by the average value. The value at the grain boundary is defined as enrichment factor [26].

With the aid of the markers, the microstructures of the AES measured area were obtained from EBSD. The microstructures and the AES results on the scanning lines are shown in Fig. 2 together.

The distribution of Nb and misorientations of the grain boundaries along the AES measured line are shown in Fig. 3. The locations of the

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