Research Square

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Quantitative Analysis of Retained Austenite in Nb Added Fe-based Alloy

Kwang Kyu Ko
Gyeongsang National University
Jin Ho Jang
Gyeongsang National University
Saurabh Tiwari
Gyeongsang National University
Hyo Ju Bae
Gyeongsang National University
Hyo Kyung Sung
Gyeongsang National University
Jung Gi Kim
Gyeongsang National University
Jae Bok Seol (jb.seol@gnu.ac.kr)
Gyeongsang National University https://orcid.org/0000-0001-9143-4274

Research Article

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Abstract

Use of Pipe lines for long-distance transportation of crude oil, natural gas etc., increasing and have pivotal importance in recent times. To improve transport efficiency through increased pressure and improved laying efficiency through reduced diameter and weight of line pipes, high specific strength plays a crucial role. TRIP-based high-strength and high-ductility alloys comprise a mixture of ferrite, bainite, and retained austenite that provide excellent mechanical properties such as dimensional stability, fatigue strength, and impact toughness. In this study, microstructure analysis performs using both Nital etching and LePera etching methods. At the time of Nital etching, it is difficult to distinctly observe second phase. However, using LePera etching conditions it is possible to distinctly measure M / A phase and ferrite matrix. The fraction measurement was done using OM and SEM images which gives similar results for the average volume fraction. Although it is possible to distinguish the M / A phase from the SEM image of the sample subjected to LePera etching, but using Nital etching it is impossible. Nital etching is good at specific phase analysis than LePera etching through the SEM images.

Introduction

Recently, the importance of pipelines as a long-distance transportation means for crude oil, natural gas, and the like is emerging. Up to now, the American Petroleum Institute (API) standard X80 or less has been applied to long-distance transport trunk line pipes. However, to improve the transport efficiency through the increase of the transportation pressure and the improvement of the laying efficiency through the reduction of the diameter and weight of the line pipes, the increase in strength is accelerating. In particular, the X120 grade line pipe, which exhibits a tensile strength of over 900 MPa and can withstand approximately twice the internal pressure of X65, can transport about twice as much gas as compared to a lower grade line pipe of the same size [1, 2].

Compared to the conventional methods of increasing the pressure enduring ability of the line pipe by increasing the pipe wall thickness, the use of high-specific strength line pipe saves the cost of materials, transportation, and on-site welding work, leading to overall construction cost savings. These enhanced properties achieved due to unique microstructure which is mainly composed of martensite/bainite mixture.

The austenite transformation structure is classified into ferrite, pearlite, bainite, and martensite [3, 4]. Martensite phase forms through diffusionless transformation when austenite phase is quenched at a very high speed. According to the carbon content, martensite can be classified into lath/plate martensite [5]. When the austenite is rapidly cooled, the carbon saturation concentration is reduced and the alloy phase supersaturated with carbon, and the deformed region has high strength due to the high dislocation density. However, since martensite has low elongation compared to strength, it is used by adding alloying elements such as manganese (Mn) and chromium (Cr) or increasing the carbon content[5].

According to the existing literature, the alloy of high strength-high ductility combination mainly developed using TRIP phenomenon reported by Zackay et al. [6]. It has ductile ferrite matrix which is strengthened by bainite which gives it excellent mechanical properties such as dimensional stability, fatigue strength, and impact toughness owing to the combination of uniform elongation generated by the transformation from austenite to martensite[7 ~ 9]. A small amount of retained austenite remains even in two-phase steels, which have been studied as high-strength-high-ductility steels since 1970s, and its reported that the ductility could be improved through transformation [10]. In the late 1980s, various studies have been conducted to improve ductility by increasing the retained austenite fraction in the structure of abnormally structured steels [11, 12]. The volume fraction, distribution of various phases, especially the transformation rate and stability of retained austenite are very important in the mechanical properties of TRIP steel [7, 9].

As mechanical properties can be determined according to the fraction of retained austenite, observation of microstructure in steel is very important. The microstructure observation of steel is generally divided into Black (pearlite, martensite), White (ferrite), Gray (martensite, bainite) phases using Nital etching. However, in steel it is difficult to accurately distinguish images due to problems of contrast, especially intermediate colors such as gray. It is well reported that LePera etching is capable of distinct phase analysis compared to Nital etching. Because LePera etching divides the phases into Blue/Green (ferrite), Brown (bainite), and White (martensite) [13–15]. According to the research result of Tsipouridis [16], cooling rate has a large influence on the martensite structure. In the case of a slow cooling rate, the inside of unrecrystallized martensite grains is transparent white while for the fast-cooling rate, the inside of the coarsened grains appeared as dark brown.

This study aims to analyze the M/A phase of Fe-Nb alloy specimens through LePera etching of the retained austenite and martensite phases which do not appear distinctly while using Nital etching.

1. Experimental

In this study, an experiment was conducted using Nb-added bainite steel (hereinafter 4Nb) according to the composition of Table 1, to observe the microstructure of API Steel. After the alloy was prepared, the specimen was reheated and maintained at 1,150° C. for 2 hours, followed by initial rolling at a rolling ratio of 56%. Thereafter, final rolling was performed at a rolling ratio of 43% at 860°C. After completing the rolling process, the specimen was cooled in water to 607°C at a cooling rate of 4.1°C/s, and then air-cooled to room temperature to prepare a specimen.

The cross section including the rolling direction and thickness direction of the specimen was subjected to marco polishing from #200 to #4000 using SiC paper, micro polishing using 3 µm and 1 µm diamond suspension. An X-ray diffraction test was performed using an X-ray diffraction analyzer (X-ray Diffraction, XRD). Cu Kα was used for the experiment, and the specimen was prepared by mechanically grinding the upper surface of the plate.

For microstructure observation, a LePera solution, which appears in various colors in the etching solution to clearly distinguish phases, was used [13, 15]. The LePera solution can be obtained from a 1:1 volume ratio of Reagent 1 and Reagent 2 as mentioned in Table 2. The condition of the etched surface by this solution depends on the amount of Reagent 1, 2. When using an optical microscope(OM), it is necessary to raise the ratio of Reagent 2 when most phases show blue and increase the ratio of Reagent 1 to make a solution when most phases show brown. The experiment was conducted based on a LePera solution prepared by mixing 100 ml of ethanol + 5 g dry picric acid + 1.5 g $Na_2S_2O_5$ + 100 ml distilled H_20, also about 4.7% Nital solution (ethanol 150 ml + Nitric acid 7.5 ml) was used for comparison with the LePera solution. The LePera solution was processed by adjusting the ratio of the mixed solution according to OM Image, and the etching time was used based on the experimental results of LePera [17] and Santofimia [18]. Thereafter, microstructure analysis was performed using an OM (Olympus BX53M) and a scanning electron microscope (SEM, JEOL (JSM-7610F)).

Table 1
Chemical composition of the alloys (wt.%)

Alloy	C	Si	Mn	P	Al	Cr	Ni	Mo	Ti	Nb	B	N
4 Nb	0.062	0.14	1.55	0.012	0.03	0.1	0.202	0.104	0.021	0.04	0.002	0.003

Table 2
Composition of LePera solutions

Reagent 1	Reagent 2
1g $Na_2S_2O_5$ 100ml distilled H_2O	4g dry picric acid 100ml ethanol

2. Results And Discussion

Figure 1 shows the XRD pattern of the 4Nb specimen. The XRD pattern analysis confirms the BCC structure only, which refers that the residual austenite that can exist as a secondary phase after transformation has a very low density which is beyond the measurement limit of XRD.

Figure 2 shows microstructure image of the specimen after Nital etching. As it can be seen from the OM image, there was a limitation in clearly distinguishing the retained austenite and martensite phases by Nital etching. To overcome the limitation of distinctly observing the phases the etching solution was changed to the LePera etching solution and the experiment was carried out. Figure 3 shows the microstructure images as per the composition mentioned in Table 3. As the composition in Table 3 are the most widely known composition, retained austenite and martensite phases were observed, but yellow/blue for ferrite at the matrix and brown color for bainite have a blurry color. Etching conditions were changed as Table 4 so that the phase could be clearly distinguished. In Fig. 4, the retained austenite and martensite phases appeared well, but the matrix was shown to be brown as a whole, which means over etched. Figure 5 was obtained by performing the experiment again by Table 5 composition. It becomes possible to clearly distinguish the retained austenite and martensite phases. Also, the fraction of the retained austenite and martensite phases could be measured with the ferrite microstructure images of the matrix. Figure 6 summarizes the volume fraction of retained austenite and martensite. It was measured to be 8–11%, and the average was 9.77%. Figure 7 shows the SEM images of specimen by Nital etching. Although there are difficulties in classifying the phases, the phase analysis was possible. Figure 9 shows the quantification of the retained austenite and martensite volume fraction obtained from the Fig. 8. In both the SEM image and the OM image, it was measured at a volume fraction of 9–11%, which is a similar result, and the average was 9.97%.

Based on the above experimental method, the volume fraction and M/A mean size were measured and compared for the 2Nb and 10Nb specimens in which only the Nb content was changed to 0.02 and 0.10 wt%. Figure 10 shows the volume fraction of M/A for 2Nb, 4Nb, and 10Nb specimens. 2Nb and 4Nb showed similar values, but the 10Nb specimen decreased by 1.5 to 2% compared to the previous two specimens. Figure 11 shows the results of measuring the mean size of M/A for three specimens. Unlike the M/A volume fraction, 4Nb and 10Nb showed similar values, but 2Nb showed lower values than 4Nb and 10Nb. A more detailed study is needed further on the correlation between the volume fraction and the mean size.

Table 3
First composition of LePera solution

No.	Etchant	Content	Condition
1	$Na_2S_2O_5$	1.0 g	30 s
2	Dry picric acid	4.0 g	
3	Ethanol (96%)	100 ml	
4	Distilled H_2O	100 ml	

Table 4
Second composition of LePera solution

No.	Etchant	Content	Condition
1	$Na_2S_2O_5$	2.0 g	30 s
2	Dry picric acid	5.0 g	
3	Ethanol (96%)	100 ml	
4	Distilled H_2O	100 ml	

Table 5
Final composition of LePera solution

No.	Etchant	Content	Condition
1	$Na_2S_2O_5$	3.0 g	30 s
2	Dry picric acid	7.0 g	
3	Ethanol (96%)	100 ml	
4	Distilled H_2O	100 ml	

3. Conclusions

Retained austenite/martensite phase transformation behavior was analyzed using a microstructure of Fe-4Nb alloy for API line pipe steel. For phase analysis at low temperature, the LePera etching method used, and analysis was performed using OM and SEM images.

1. LePera etching was used to classify low-temperature phases (Bainite, Martensite), where Bainite was classified as Brown and Martensite as White. In particular, the martensite grain boundaries were uniformly

distributed over the entire surface and mixed as white areas.

2. LePera etching conditions varies, depending on the steel composition. In this study, we found the most suitable etching condition of the specimen, $Na_2S_2O_5$ 3.0g, dry picric acid 7.0g, and holding time 30s.
3. During Nital etching, the representative components constituting the microstructure, i.e., grain boundary ferrite, widmanstaten ferrite, acicular ferrite, etc., are clearly distinguishable and useful for quantitatively measuring each fraction, but the Martensite/Austenite (MA) phase is indistinguishable.
4. Similar results were obtained when measuring fractions in OM and SEM during LePera etching.
5. The M/A phase could be observed on the OM image through LePera etching, and since it is difficult to obtain various information from the SEM image analysis, Nital etching is suitable for observing various tissues.

Declarations

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Authors' contributions

Kwang Kyo Ko: Data curation, Formal analysis, Investigation, Writing – original draft. **Jin Ho Jang**: Data curation, Formal analysis, Investigation, visualization. **Saurabh Tiwari**: Investigation, English proceeding. **Hyo Ju Bae**: Formal analysis, Investigation. **Hyo kyung Sung**: Writing – review & editing. **Jung Gi Kim**: Writing – review & editing. **Jae Bok Seol**: Conceptualization, Project administration, Writing original draft, Writing – review & editing.

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Availability of data and materials

All relevant data have been included in the manuscript itself.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Figures

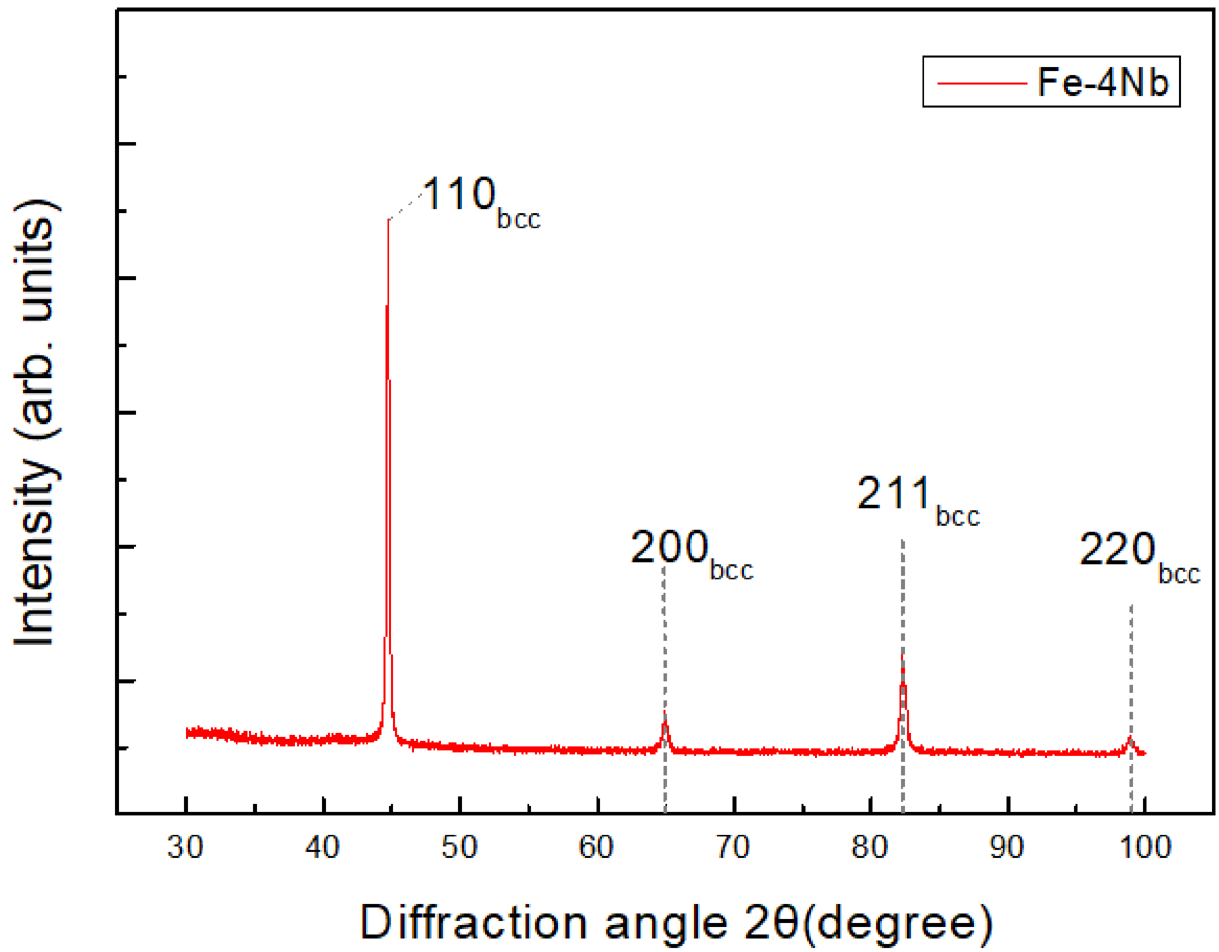

Figure 1

XRD analysis for measuring the retained austenite fraction

Figure 2

Optical micrographs of Nital etching

Figure 3

Optical micrographs of Primary condition of LePera etching

Figure 4

Optical micrographs of Second condition of LePera etching

Figure 5

Optical micrographs of Final etching conditions of LePera etching

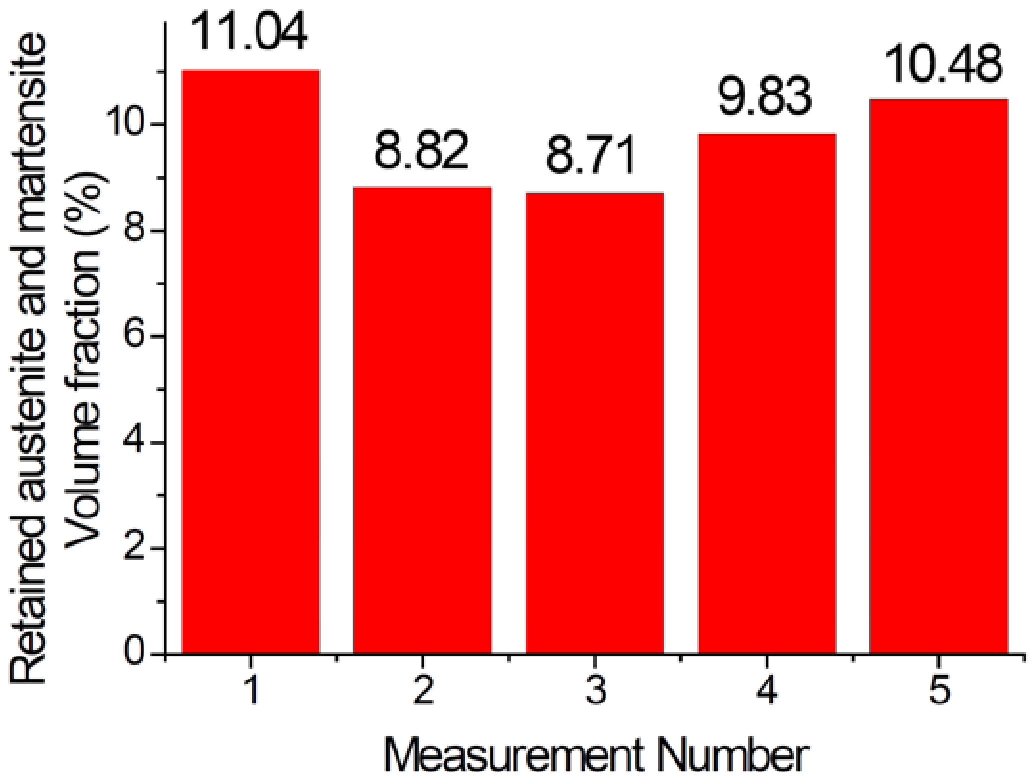

Figure 6

Results of volume fraction measured by OM for LePera etching

Figure 7

SEM micrograph of Nital etching

Figure 8

SEM micrograph of LePera etching

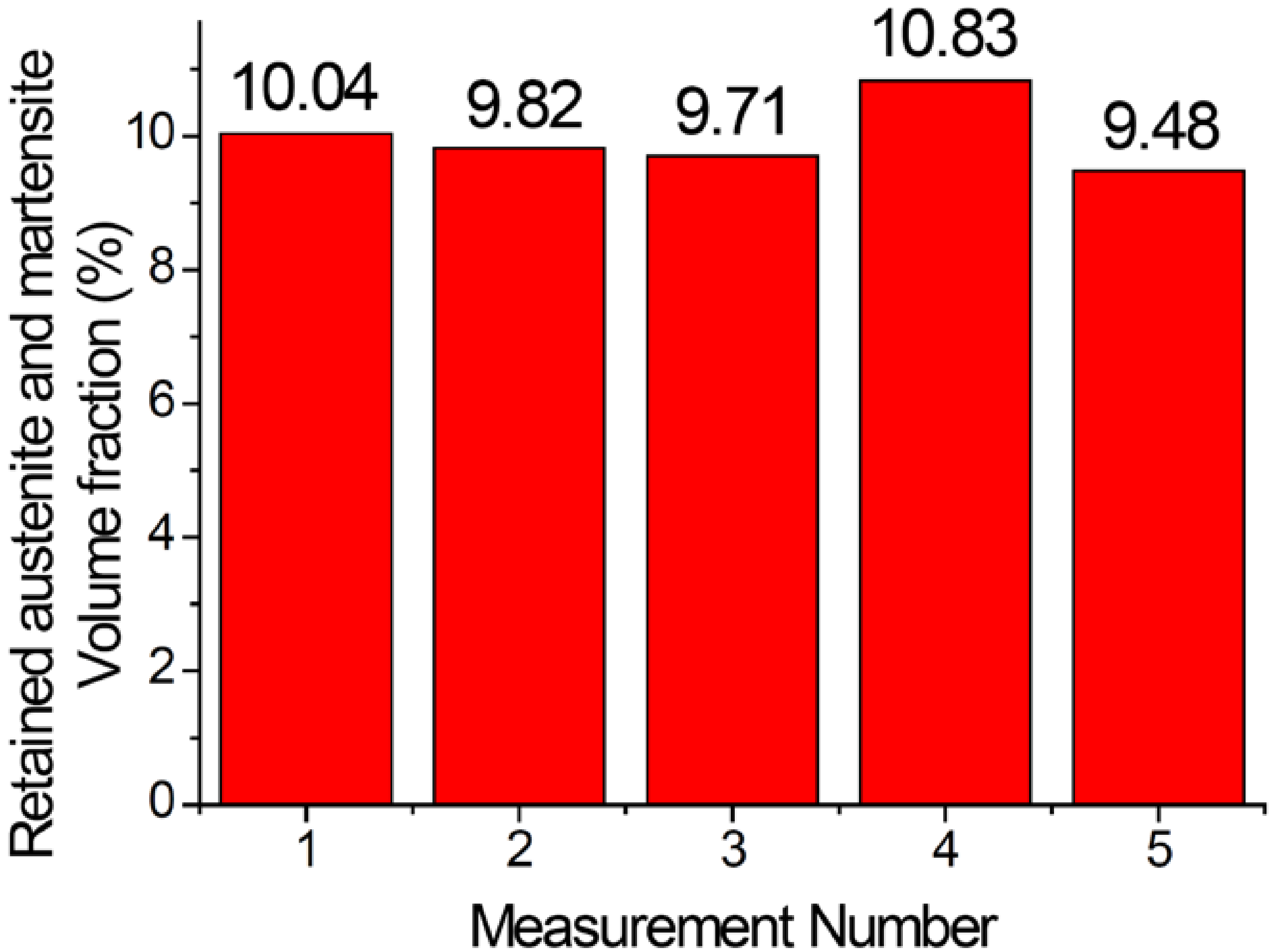

Figure 9

Results of volume fraction measured by SEM for LePera etching

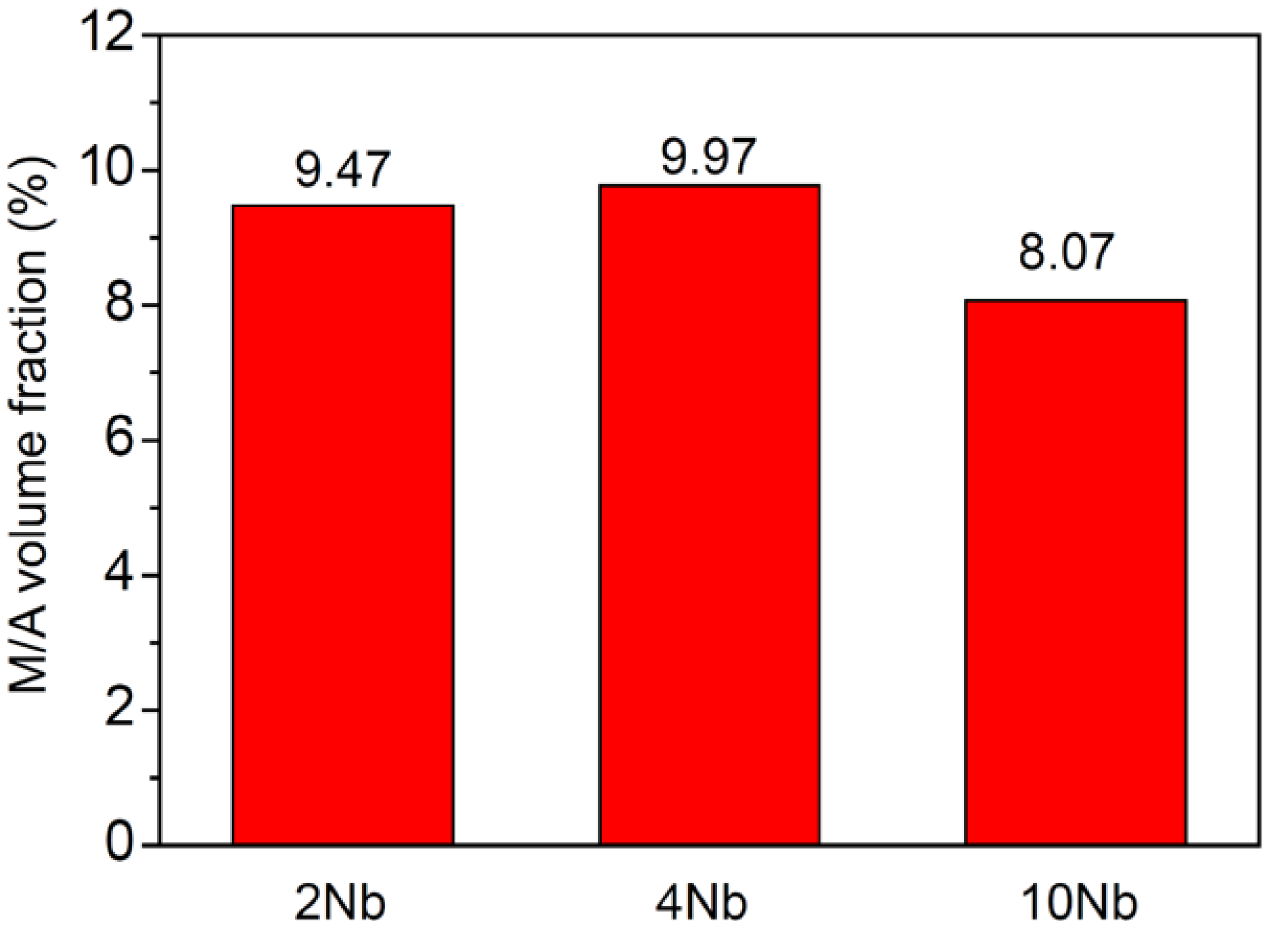

Figure 10

Results of volume fraction based on different Nb contents

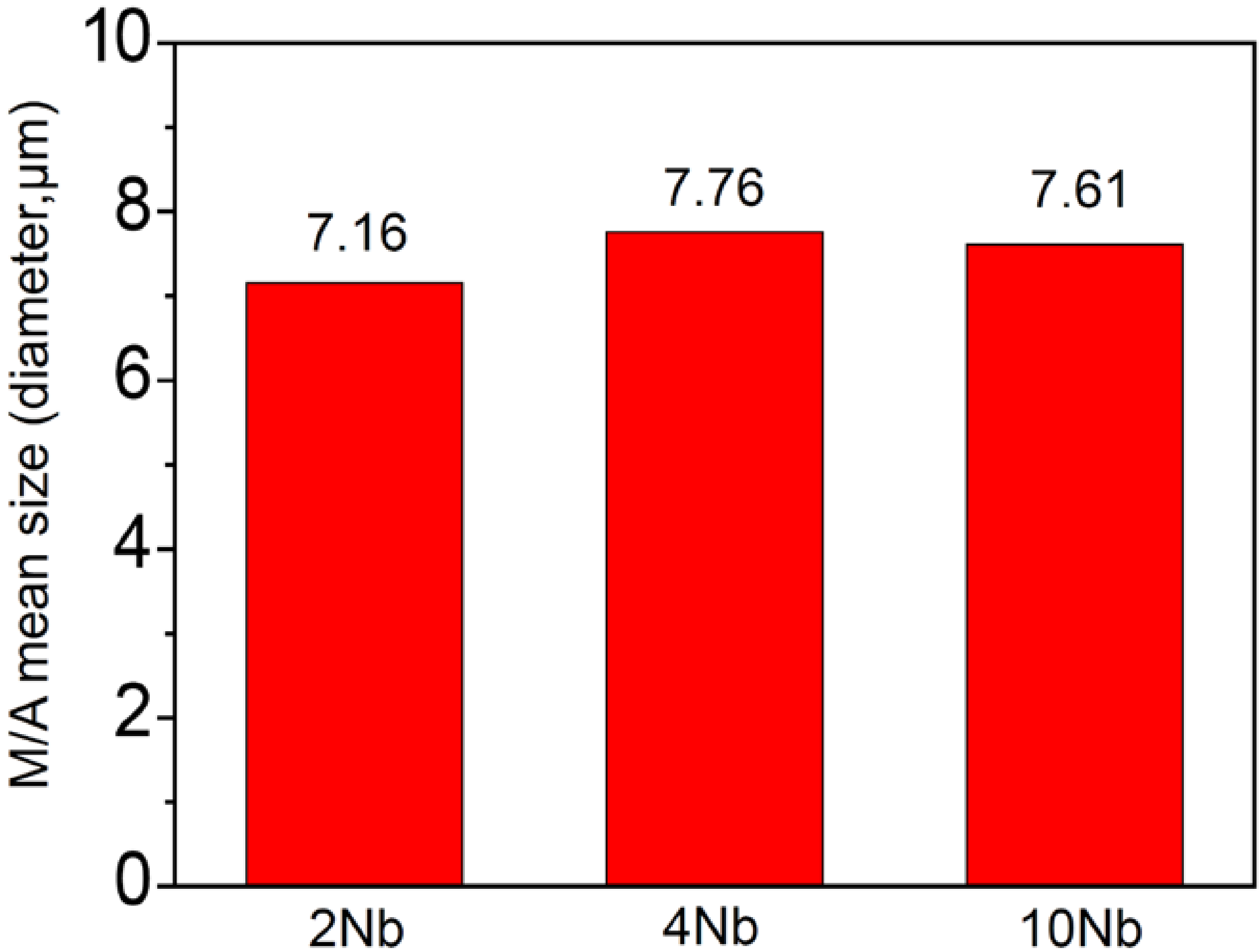

Figure 11

Results of M/A mean size based on different Nb contents