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Analysis of the Distribution of Light Elements in Steels by Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)

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Abstract

Analysis of the distribution of light elements with concentrations on the order of partsper-million (ppm) in complex microstructure of steels is a key requirement for the understanding and control of microstructures and resultant mechanical properties of advanced high strength steels. In this paper, ToF-SIMS analysis was performed to investigate the distribution of boron and hydrogen isotope in steels. A Ga Focused Ion Beam (FIB) was used as a primary ion source for high spatial resolution analysis. It was demonstrated that Ga-FIB-ToF-SIMS is a very powerful tool to analyze the distribution of light elements in steels at the sub- micrometer scale, which is (almost) impossible for other characterization techniques such as Electron Probe Micro Analysis (EPMA).

1. Introduction

Steel materials, by necessity or design, contain carbon and various other elements in iron, the main component of steel. These elements are distributed in microstructures in the course of manufacturing steel or in the environment in which the steel is used. Advanced control of elements distribution can significantly contribute to the final microstructure and the resultant improved mechanical properties of steel materials. For instance, in the case of high-tensile steel plates, the morphology and volume fraction of hard martensite (M) phase is highly controlled.¹⁾ The hardenability, which represents the likelihood of the M phase developing, can be remarkably improved by adding only 10 ppm of boron (B).²⁾ The hardenability improvement is brought about by B segregated at the austenite (γ) grain boundaries suppressing the ferrite (α) phase formation from the γ grain boundaries during cooling. However, when B and coexisting elements together form precipitates, the amount of B that is segregated at the γ grain boundaries decreases, making it difficult to obtain the desired hardenability. Given this, there is a demand for an analytical method to obtain parameters for the control of the distribution (solid solution, precipitation, and segregation) of B in steel materials

As with B, hydrogen at the several ppm level, which causes hydrogen embrittlement in high-tensile steel plates, has posed a pressing task of understanding the hydrogen trapping behavior in microstructures.³ Regarding phosphorus and sulfur as well, several dozen ppm of one of the two can be segregated at grain boundaries to cause the grain boundary embrittlement. In order to prevent the grain boundary embrittlement, it is necessary to clarify the relationship between the distribution of these elements and the processing conditions/material composition.^{4,5)} Nevertheless, there is no universal quantitative hydrogen distribution analysis technique that has been established to date; and the detection of a light element at the several dozen ppm level by conventional analysis techniques, such as electron probe micro analysis (EPMA) is difficult. If a technique to characterize the distribution of a slight amount of a light element is developed, then new guiding principles may be discovered for developing high-tensile steel plates and the manufacturing processes.

This article reports the results of our study using time-of-flight secondary ion mass spectrometry (ToF-SIMS) to characterize the distribution of light elements present in steel in small amounts. The secondary ion mass spectrometry (SIMS) allows for analyzing hydrogen and isotopes and is suitable for analyzing minor elements because of the extremely small background in mass spectra. In order to observe steel microstructures with high spatial resolution, we used a gallium focused ion beam (Ga-FIB) as a primary ion beam source. The system for this was developed during the project led by the Japan Science and Technology Agency called the Development of Advanced Measurement and Analysis Systems, in which Nippon Steel Corporation participated in the past. The system has achieved the highest lateral resolution of 40 nm for secondary ion images. For

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the detailed configuration of the system, please see the references^{6,7} listed at the end of this article. We will describe the cases in which the system was used to characterize the distribution of B and hydrogen isotopes in steel.

2. Need for High Spatial Resolution and Challenges

Analysis to clarify element distribution requires sufficient high spatial resolution to observe microscopic precipitates and the boundary segregation. This chapter discusses the spatial resolution required to analyze the boundary segregation. Figure 1(a) is a schematic diagram showing the primary probe irradiation onto a grain and just on a grain boundary in a SIMS analysis. The width of grain boundary segregation is assumed to be the thickness of an atomic layer; the concentration of a segregated element in an atomic layer of the grain boundary is β times the concentration of the same element in the grain (β : segregation coefficient). As shown in Fig. 1(b), the ratio between the number of elements captured in the primary probe in the grain boundary and that in the grain is a function of the segregation coefficient and the primary probe diameter. The smaller the probe diameter becomes and the larger the segregation coefficient becomes, the difference of the element concentration between the grain boundary and the grain interior becomes more substantial. When using a smaller probe diameter, elements with smaller segregation coefficients can be observed.

Although Ga-FIB-ToF-SIMS has the highest lateral resolution of





40 nm, the beam required for small amount element analysis is several hundred nm in diameter. This makes it difficult for Ga-FIB-ToF-SIMS to observe grain boundary segregation when the segregation coefficient is between 10 and 100. According to Seah, the smaller the solid solubility limit an element has, the more the grain boundary segregation of the element is likely to take place, and Ni, Mn, and other elements with large solid solubility limits have the segregation coefficient below 100.⁸ Elements with the segregation coefficient exceeding 100 include B, S, C, N, P with small solid solubility limits. For these elements, the grain boundary segregation can be detected using the spatial resolution of SIMS.

However, it is difficult to analyze N, for which its secondary ion yield is very low, and C, which is susceptible to the surface contamination layer. P and S, the mass of which can cause interference with SiH and O_2 , respectively, require high mass resolution (approx. $m/\Delta m$ to 3000 for separating S and O_2 , where *m* is mass) to separately detect them. For the above reasons, elements that can be analyzed for grain boundary segregation using SIMS are extremely limited. In addition, the signal intensity decreases in compensation for high spatial resolution analysis, and the secondary ion yield improvement achieved when using a Cs or O_2 primary ion beam is not recognized when using Ga-FIB. This constitutes challenges required to be overcome in order to use Ga-FIB-ToF-SIMS.⁹

3. Application to Distribution Analysis for Small Amounts of Elements Contained in Steel

3.1 Analysis of boron in steel¹⁰⁾

In addition to electron energy loss spectroscopy¹¹⁾ in a scanning transmission electron microscope (STEM) and atom probe¹²⁾, SIMS capable of mapping wider fields has been used^{10, 13–16)} for the characterization of B segregated at grain boundaries in steel. Since the amount of an element segregated at grain boundaries in general depends on grain boundaries within a field of view is useful in understanding the entire B distribution picture. However, given the situation with the challenge regarding decreased signal intensity when a high spatial resolution Ga-FIB is used for the primary ion beam, we examined optimal analytical conditions using our instruments.

The accelerating voltage for the Ga-FIB was set to 30 kV. The test piece was irradiated with the primary ion beam at a frequency of 15 kHz with a pulse width of 200 ns during the SIMS analysis. Maps were obtained when the test piece was scanned using the Ga-FIB primary ion beam. The number of pixels on a map was 128² or 256², and the irradiation was 200 pulses per pixel. As described later, we removed the contaminated surface layer that consisted of hydrocarbon using a Ga DC ion beam prior to the SIMS analysis. As secondary ions derived from B, we considered ¹⁰B⁺ (mass-to-charge ratio m/z=10), ¹¹B⁺ (m/z=11), ¹¹B¹⁶O⁻ (m/z=27), and ¹¹B¹⁶O⁻₂ (m/z=43).

We prepared steel material samples containing 10, 15, and 30 ppm of B, all of which contained M structures. The surface mirrorpolishing is sufficient for conducting an SIMS analysis, and special pretreatment is not necessary. However, for observing microstructures using the channeling contrast when a secondary electron image taken by ion beam irradiation is examined, the surface may be treated by colloidal silica polishing or Ar ion beam sputtering. In such a case, the electron back scatter diffraction (EBSD) measurement and SIMS analysis can be performed in the same region.

Figure 2 shows the secondary ion map images of a steel material that contains 30 ppm of B. The images show that B existed as a pre-



Fig. 2 (a) Secondary electron image, (b)–(e) Secondary ion image obtained from the 30-ppm boron containing steel (b) m/z=10, (c) m/z=11, (d) m/z=27, (e) m/z=43

cipitate when any B-derived secondary ion was used. The signal intensity ratio for m/z=10 and m/z=11 shown in Fig. 2 (b) and (c) is 1:3.9, which is nearly equal to $1:4^{17}$, the natural isotopic abundance ratio of ¹⁰B and ¹¹B. For m/z=27 and m/z=43, the B distribution is basically the same as that shown in Fig. 2 (b) and (c), except that in the secondary ion map shown in Fig. 2 (e) for m/z=43, streaks of B were observed along grain boundaries in addition to B precipitates. In view of the fact that the system used for the analysis was equipped with a time-of-flight mass spectrometer and also that this was a top surface analysis, possibly with the aid of the signal intensity of molecular ions composed of B and oxygen (O) derived from a natural oxide that might have been higher than the signal intensity of the atomic ions of ¹⁰B⁺ or ¹¹B⁺, the grain boundary segregation was able to be captured in the map.

Then, we investigated the dependency of the secondary ion intensity on the depth direction using a steel material containing 15 ppm of B shown in **Fig. 3**(a), while sputtering the native oxide on the surface. As the time period of pre-sputtering increased, the secondary ion intensity of ¹⁶O⁻ decreased, accompanied by a decrease of the secondary ion intensity of m/z=43. For this reason, it was necessary to analyze the secondary ion for m/z=43 in the presence of surface oxygen. However, in the case in which pre-sputtering was not performed at all, although the signal intensity for m/z=43showed its maximum, the B distribution was not able to be captured as shown in Fig. 3 (b). The B distribution was observed with clarity when pre-sputtering was performed for 10 to 20 seconds as shown in Fig. 3 (c) and (d) in which B can be seen being distributed along grain boundaries.

In addition to ${}^{11}\text{B}{}^{16}\text{O}_2^-$ (m/z=42.999), ${}^{12}\text{C}_2^-\text{H}_3^{-16}\text{O}^-$ (m/z=43.018) could be a candidate for secondary ions for m/z=43. On the outer-

most surface, secondary ions derived from the contaminated layer consisting of hydrocarbon systems were likely to bring about mass interference with secondary ions derived from B. In fact, m/z=24 (${}^{12}C_{2}^{-}$) derived from carbon (C) was observed with the highest secondary ion intensity on the outermost surface. Incidentally, considering that the secondary ion intensity for m/z=24 became stable after 50 seconds had elapsed during sputtering, it is desirable to remove any natural oxides on the surface in order to analyze C in steel. From the examination described above, it has been revealed that an optimal surface condition for analyzing B or C does exist, and that it is important to adjust the sputtering time depending on the element to be observed.

Figure 4 shows the B distribution (in a steel material containing 10 ppm of B) obtained by adjusting the surface condition. Although the observed grain boundary segregation width is approx. 200 nm, the actual segregation is about as wide as the thickness of several atomic layers. Therefore, the observed width can correspond more or less to the diameter of the beam. By appropriately selecting the secondary ion species and by adjusting the surface condition as described above, the grain boundary segregation and precipitates can thus be characterized in the order of sub-µm using our Ga-FIB-ToF-SIMS system. In the meantime, while the grain boundary segregation of B is clearly observed in Fig. 4 (for a steel material containing 10 ppm of B), many precipitates are shown in Fig. 2(e) (for one containing 30 ppm of B). Given this, the behavior of B regarding grain boundary segregation and precipitation depends on the amount of B in the steel. Other factors that exert significant influence on the behavior of B are coexisting components and heat treatment conditions.^{2, 11, 13, 16, 18)} The Ga-FIB-ToF-SIMS analysis has been confirmed to be capable of relatively short-time analysis as it only took 10 to



Fig. 3 (a) Sputter depth profile of secondary ion of m/z=16, 24 and 43, (b)–(g) Secondary ion maps of m/z=43 at a sputtering time of (b) 0s, (c) 10s, (d) 20s, (e) 30s, (f) 40s and (g) 100s



Fig. 4 Boron distribution in the 10 mass-ppm boron containing steel

20 minutes to complete B mapping per field of view; and it required only simple sample preparation, allowing a larger number of fields to be analyzed. Hence, a Ga-FIB-ToF-SIMS analysis has been demonstrated to be a practical tool for understanding the B distribution in steel materials that contain B and the dependency on the steel composition and heat treatment.

3.2 Analysis of hydrogen isotopes in a Fe-30%Ni alloy¹⁹⁾

In order to reveal the mechanism of hydrogen embrittlement, which is particularly problematic with high-tensile steel, and to seek countermeasures, there is strong demand for the analysis of hydrogen penetration behavior into steel and hydrogen distribution behavior in microstructures. In principle, hydrogen and its isotopes cannot be directly observed by electron or X-ray spectroscopy, and so are analyzed by mass spectrometry.²⁰⁾ Still, analyses are being conducted in the situation without analytical techniques and conditions having yet to be established. For the techniques to observe hydrogen distribution currently used, please refer to the article by Akiyama²¹⁾. Below, we describe a case in which an Fe-30%Ni model alloy was charged with deuterium (D), a hydrogen isotope, and the distribu-

tion of D was characterized using our Ga-FIB-ToF-SIMS system, for studying the conditions for characterizing the hydrogen distribution in steel.¹⁹⁾ D was used instead of hydrogen, because residual hydrogen gas inside the SIMS chamber is left on the steel plate surface after sputtering and constitutes a background, making it difficult to obtain net hydrogen secondary ion and its distribution.

The Fe-30%Ni alloy was prepared by vacuum melting and then undergoes the thermo-mechanical processes and heat treatment. Although the alloy has the phase of γ , some samples were cooled after the D charging to obtain a two-phase structure consisting of M and γ . For the D charging, a solution was prepared by adding 3%NaCl and ammonium thiocyanate to heavy water, and each sample was set as a cathode and was electrolytically charged with D. Pt was used for an anode. The sample used to characterize the diffusion behavior of D during the D charging was electrolytically charged with D after partly masking the sample as shown in Fig. 5(a); Masks were placed on the top and bottom faces of the sample thereby allowing D to enter from the sides, and were removed immediately after the completion of charging to characterize the diffusion behavior of D using the Ga-FIB-ToF-SIMS as shown in Fig. 5(b). The Ga beam irradiation conditions here were basically the same as those applied when characterizing the distribution of B. As the secondary ions derived from D, we selected m/z = 18 (¹⁶O²D⁻).

3.2.1 Characterization of deuterium distribution in a M-y dual-phase alloy

Figure 6(a) shows the distribution of secondary ion intensity at m/z=18 obtained from the surface of the M- γ dual-phase alloy. As seen from the figure, D is heterogeneously distributed. The secondary ion intensity obtained from the γ phase was greater than that from the M phase. This is because the hydrogen solid solubility in the γ phase is higher. At the same time, a D-deficient region was ob-

served in the area from the interface between the γ and M phases to part of the γ phase across the interface. Although the reason for this is uncertain, here is our hypothesis: After the D charging, D is released from the surface. D is released at a greater rate from the surface of the M phase with a larger diffusion coefficient. As a result, D in the γ phase close to the M phase is absorbed to the M phase, forming the region deficient in D.

The reason for the secondary ions of D observed on the surface of the M phase then, as well as from the γ phase, is likely to be the D flux from the outermost surface of the M phase that was able to be observed since D had sufficient time to diffuse well within the time allowed by the Ga-FIB pulse width of 200 ns. In order to conduct an analysis in a more detailed manner, it is necessary to examine the time elapsing while D is distributed after the D charging. To this end, we consider it indispensable to use a cooling mechanism to freeze the D distribution in a test piece after the D charging is completed until the test piece undergoes an SIMS analysis. The D distribution is also possibly affected by the elastic/plastic strain of surrounding γ as a result of the M phase transformation.²²⁾ Although further consideration is necessary, the use of a Ga-FIB-ToF-SIMS system has definitely made it possible to directly observe the characteristics such as the D-deficient region in the vicinity of the interface between different phases.

Figure 6(b) shows the D distribution obtained by heating the sample to 130°C and held for 100 minutes after the process described above performed to obtain Fig. 6(a). By heating, the D distribution on the surface was no longer visible. After being left in the SIMS chamber at a room temperature for one hour, the sample was measured again. Figure 6(c) shows the result. The D distribution was visible again, but this time, D was observed on the M phase surface rather than on the γ phase surface. This indicates that while





Fig. 5 Schematics of fcc Fe-30%Ni specimen for (a) Deuterium charging and (b) SIMS analysis



Fig. 6 Secondary ion maps of m/z = 18, ¹⁶O²D⁻

(a) 24h after charging finished; and (b) 0.5h and (c) 1.5h after the completion of 100 min of annealing at 130°C, ' γ ' and 'M' in the figures represent austenite and martensite, respectively



Fig. 7 Circle line profile of secondary ion of m/z=18, ¹⁶O²D from γ Fe-30%Ni alloy. Dotted line fitting results.

the heat treatment caused D to dissipate from the surface, D remained in the interior of the sample and spread back to the surface again during the retention at room temperature. The diffusion coefficient of D is larger in the M phase than the γ phase when D diffuses from the interior to the surface of a sample. Given this, it is considered that the M phase became a diffusion path, and the larger secondary ion intensity of D was recognized on the M phase surface. Thus, the SIMS analysis can be an effective means to observe nonuniform behavior of hydrogen diffusion in a multi-phase structure. 3.2.2 Visualization of deuterium diffusion in the γ phase

Figure 7 shows the profile of m/z=18 obtained by charging a single phase γ Fe-30%Ni alloy masked in the manner shown in Fig. 5 (a) with D, removing the mask layers to perform a SIMS analysis, and having the alloy undergo the surface line scan (Fig. 5 (b)). This corresponds to the depth analysis (diffusion profile) of D. Normally, when performing depth analysis, the secondary ion intensity is recorded while sputtering the surface using an ion beam. With this method, along with an increase of the sputtering time, surface roughness progresses, constituting an issue of the depth direction resolution deteriorating over time. By performing a line scan for the depth direction analysis as shown in Fig. 5, the D diffusion profile can be obtained, while avoiding deterioration of the depth direction resolution.

As shown in Fig. 7, the secondary ion intensity of D decreased on the outermost surface, and D partly dissipated from the surface during the period from the completion of charging to the start of the SIMS analysis. The dotted-line profile in Fig. 7 is the result of curve fitting using the equation of bulk diffusion²³⁾ for the profile of experimental data. In the part of the figure indicated by the arrow, the secondary ion intensity is higher with the experimental data than with the curve created by fitting the experimental data. This suggests that a short-circuit diffusion path of D existed.

The first-principles calculation performed by Du et al.²⁴⁾ demonstrated that grain boundaries did not provide short-circuit diffusion paths of hydrogen in γ -Fe. On the other hand, in Ni²⁵⁾ and Fe-Mn-C austenitic steel²⁶⁾, the existence of short-circuit diffusion paths of hydrogen and D was confirmed using the hydrogen microprint technique and SIMS. However, due in part to the spatial resolution issue of SIMS, there has not been a case so far that reports on the direct observation of short-circuit diffusion paths of (heavy) hydrogen using SIMS.

Figure 8 shows the result of diffusion profile mapping conducted



Fig. 8 Secondary ion map of m/z=18, ¹⁶O²D, from fcc Fe-30%Ni alloy

through our Ga-FIB-ToF-SIMS in view of the situation. From the figure, D can be seen to diffuse from the surface to the interior of the test piece. D distributed along grain boundaries has been recognized as well. This means that the direct visualization of grain boundaries constituting high-speed diffusion paths in a γ -phase Fe-30%Ni alloy was achieved. The diffusion distance is considered to relate to the characters and linkage of grain boundaries. Conducting such detailed tests is likely to be possible through our Ga-FIB-ToF-SIMS that comes with high spatial resolution.

4. Prospects of SIMS Analysis

The use of SIMS with high spatial resolution allows for the characterization of the behavior of B involving grain boundary segregation and precipitation, and for the observation of a layer deficient in D and diffusion of D along grain boundaries. The behavior of these elements that involves the phenomenon in the order of sub- μ m has been difficult to observe in the past. However, the application of SIMS to the analysis of light elements contained in small amounts in steel has not yet been fully developed. The following issues must be resolved in order to move the SIMS analysis advancement one step further.

The first issue is the difficulty in the quantitative analysis. ToF-SIMS is extremely susceptible to the surface condition and is readily affected by the amount of oxygen on the surface and the surface layer that is contaminated. In addition, the secondary ion yield widely varies depending on whether an element that is the analyte is mixed with another element to form a solid solution or forms precipitates as a result of matrix effects. There are growing expectations for the use of contamination traps and an analytical technique that involves post-ionizing sputtered neutral species²⁷⁾ in overcoming the issue.

Secondly, hydrogen analysis is faced with an issue of hydrogen dissipation, particularly in the case of a crystal structure that consists of the α phase with a large hydrogen diffusion coefficient. Presumably, it is necessary to study a cooling mechanism to freeze the hydrogen distribution after charging. Another countermeasure is a recently considered one involving charging with hydrogen inside the SIMS chamber.²⁸⁾

Other countermeasures to tackle the difficulties include the upgrade of basic performance of instruments such as the mass resolution and detection efficiency, which is expected to help SIMS be applied to the distribution characterization of S and P as well.

5. Conclusion

This article presented case examples of the application of Ga-FIB-ToF-SIMS, which was originally developed in the national JST project in which Nippon Steel participated, to the distribution characterization of light elements contained in a small amount in steel

materials. The SIMS with high spatial resolution produced some positive results, including the direct visualization of segregation and diffusion of elements at grain boundaries. Regardless of the easy-touse sample preparation, SIMS is an analytical technique susceptible to the surface condition, requiring that caution be used when setting the conditions for analysis.

In the future, we will strive to improve the basic performance of the SIMS system in order to use it for quantitative analysis and to expand the range of elements that can be handled. Combining with analytical tools that allow atomic-level observation such as a STEM and atom probe, we will work on thoroughly illuminating the distribution of elements in steel materials at multi-scale in pursuit of contributing to the development of new steel materials and the manufacturing processes.

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