Extended Abstract of PSA-19

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ULV-SEM-EDX analysis of fine precipitates in Cr-Mo steel using windowless silicon-drift detector

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Elemental analysis technique using a latest windowless silicon-drift detector (SDD) and ultra-low voltage scanning electron microscope (ULV-SEM) have been applied to a Cr-Mo added steel containing several types of carbides and aluminum nitride. Low primary electron energy of around 1 keV reduces interaction volume and then provides remarkably high spatial resolution for x-ray analysis. We obtain elemental mappings of the precipitates with almost the same spatial resolution as ULV-SEM imaging.

1. Introdiction

The mechanical and other properties of steels are greatly influenced by the material microstructure. Especially, precise control of nanometer sized precipitates enables dramatic improvement of mechanical properties. Evaluation of such precipitates is important to design steels with higher performance. We developed a new method of identifying precipitates in a 2.25Cr-1Mo steel based on the relationship between contrasts in the ultra-low scanning electron microscope (ULV-SEM) images and chemical composition evaluated by an energy dispersive x-ray spectrometry (EDX) equipped with a field-emission scanning transmission electron microscope (FE-STEM) [1]. In this work, we tried to distinguish the precipitates by high spatial resolution SEM-EDX analysis for the bulk specimen of the 2.25Cr-1Mo steel. SEM-EDX analysis in the usual condition has not been commonly used owing to the great influence of the matrix due to poor spatial resolutions. We have used a latest windowless silicon-drift detector (SDD) [2] under the low primary energy conditions. Elemental mappings and EDX spectra are presented and discussed.

2. Experimental methods

A 2.25 mass% Cr - 1 mass% Mo steel containing several types of carbides and aluminum nitride (AlN) was used. A mirror-polished cross section was prepared for the analysis. The details in the sample preparation were reported in the previous paper [1]. A Schottky field-emission SEM system (ULTRA 55, Carl Zeiss Microscopy) and EDX system (Ultim Extreme, Oxford Instruments) were used in this study. The latter is a windowless EDX system with a 100 mm^2 SDD sensor. One can detect low energy x-ray down to a few tens electron volt under the relatively short working distance (WD) thanking to the design of the system.

3. Results and discussion

Fig. 1 (a) and (b) show the typical in-lens backscattered electron (BSE) images of precipitates recorded with the primary electron energy of 1 kV and WD of 5 mm. In Fig. 1 (a), 3 types of precipitates i - iii exist. Type ii precipitates can be differentiated from matrix only by the in-lens SE imaging (not shown here) [1]. Fine precipitates (iv) were observed in the high magnification image in Fig. 1(b). Based on previous work [1], precipitates numbered by i, ii, iii, and iv in Fig. 1 are identified to M_6C, $M_{23}C_6$, AlN, and M_2C, respectively.

Fig. 2 shows the elemental mapping data obtained using an accelerating voltage of 1.5kV for the same areas as those shown in Fig.1. All precipitates observed in Fig.1 are visualized with almost the same spatial resolution as elemental distributions. M_6C, $M_{23}C_6$, and AlN are clearly distinguished by the intensity distribution of Mo, Cr, and N x-rays, respectively in Fig.2 (a). It is noted that composite precipitates consisting of two or three components are detected

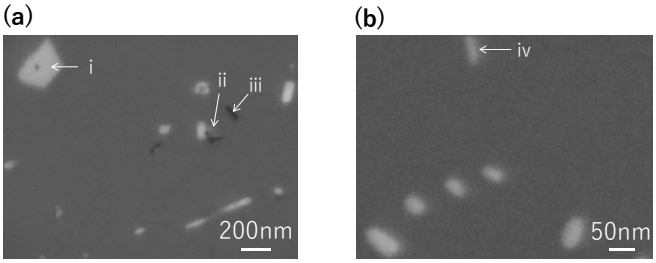

Fig. 1 Typical in-lens BSE images of the precipitates in 2.25Cr-1Mo steel at 1 kV with a WD of 5 mm.

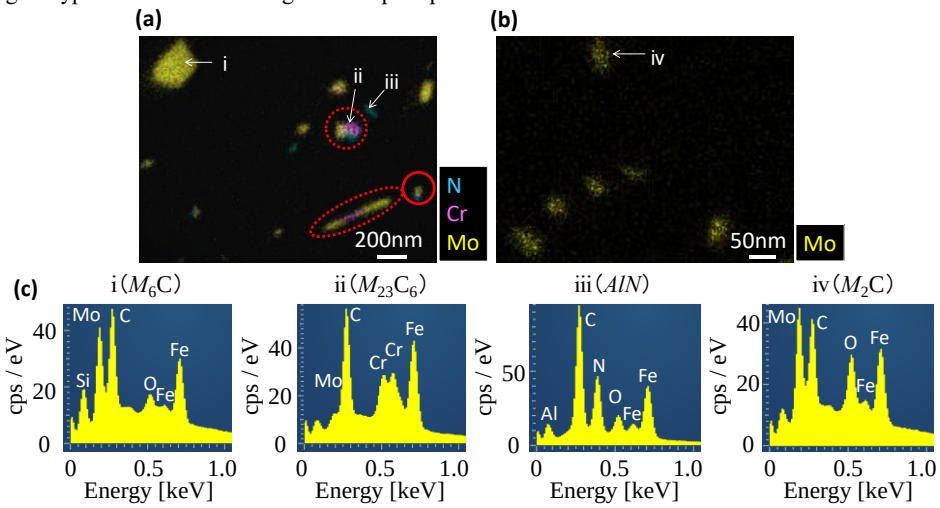

Fig. 2 (a)-(b) The composite EDX layered element map at 1.5kV with a WD of 5mm at the identical areas of Fig. 1 (a) and (b) respectively. (c) EDX spectra of precipitates i –iv in Fig. 1 and Fig. 2 at 1.5kV with a WD of 5mm

(dashed circles) and an AlN which is unclear in the ULV-SEM image is seen as noted by a solid circle. Fine precipitates with the width of approximately 15 nm (Fig. 1 (b) iv) are visible by the Mo map in Fig.2 (b).

Fig. 2 (c) shows the EDX spectra of type i - iv precipitates measured with an accelerating voltage of 1.5 kV. Four precipitates are characterized by the x-ray intensities as follows; M_6C: Si(L-line) and Mo(M-line) $M_{23}C_6$: Cr(L-line), AlN: N(K-line) and Al(L-line), M_2C: Mo.(M-line). Detection of Al(L) and Si(L) are impossible when usual EDX systems are used, which means that separation between M_6C and M_2C are difficult.

We show the fine precipitates in steel material are imaged and identified by ULV-SEM-EDX analysis by detecting low energy x-rays using the windowless SSD system. This is a big advantage of the technique as well as thin surface analysis [3]. The spatial resolution roughly estimated from Fig.2 is less than 20 nm. This value is better than from 50 nm [4] to 300 nm [5] reported for FE-scanning Auger electron microscopy (FE-SAM). Although the spatial resolution of the ULV-SEM-EDX is poor when compared with STEM-EDX, the technique is advantageous at the view point of wide-area and bulk-specimen analysis. This is thus an important technique complementing FE-SAM and STEM-EDX for fine particles in matrix.

4. Conclusion

We have demonstrated that elemental analysis with the primary electron energy of 1.5kV allowed almost the same high spatial resolution as ULV-SEM imaging for precipitates in steel matrix. Micro-analysis with the ULV-SEM-EDX is applicable in practical use and will provide crucial solutions in the many industrial fields.

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5. References

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