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学 位 （ 博 士 ） 論 文 要 旨
(Doctoral thesis abstract)

<table>
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<tr>
<td>論文題目
Title</td>
<td colspan="5">Development of analysis method of the size distribution and dispersion control in a liquid phase for nano-precipitates in steels</td>
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論文要旨（和文要旨(2000 字程度)または英文要旨(500words)）
※欧文・和文どちらでもよい。但し、和文の場合は英訳を付すこと。
Write a summary in Japanese (2000 characters) or in English (500words).
If the abstract is written in Japanese, needed to translate into English.

　　Formation process of fine nanoparticles and sub-micrometer scaled particles which are called as precipitate in steel are used for the control of functional behavior of various steel sheets. For high-tensile strength steel, fine precipitates under 10 nm are mainly dispersed in crystal grains of steel. For a steel plate, sub-micrometer sized precipitates are generated at grain-boundary. It is difficult to measure the size distribution of these precipitates in steel due to the non-uniform distribution in the steel sheets and the co-existence of small and large particles with different chemical compounds. Conventionally, the image processing of electron microscope observation has been used for the size distribution measurement, however, for the quantitative analysis of the size and chemical compound distribution it is necessary to require a lot of images and spend for long time observation. New advanced characterization method needs to develop for the rapid characterization of nanometer scaled precipitate in steel.

　　The major aim of this study is to develop the analytical method for the macroscopically characterization of these precipitates and pre-treatment method for extraction of precipitates in steel samples for the above subjects. The selective potentio-static etching by electrolytic dissolution (SPEED) method are used for the extraction of the precipitates in steels into organic solution. The asymmetric flow field-flow fractionation (AF4)-inductively coupled plasma-mass spectrometry (ICP-MS) system are conducted to measure size and chemical compound distribution of precipitates in a liquid phase. Since the dispersion control techniques are required for the determination of actual size information of nano-precipitates, the combination of dispersants, solvents and particles has to be considered for the avoidance of mass spectral interference in ICP-MS. Furthermore, the chemical reactivity between particles and other matrices in liquid phase is carefully to be escaped during the process of the pre-treatment and size measurement.

In this work, firstly, the accuracy of AF4 analysis was investigated by the comparison among transmission electron microscope (TEM) and small-angle X-ray scattering (SAXS), using gold

nanoparticles. The peak broadening detected by AF4 was discovered and the correction method for size distribution was developed. Next, the scope of application of AF4-ICP-MS was expanded by the improvement of sample introduction efficiency using the mistral-desolvating system. Thirdly, the detection sensitivity of AF4-ICP-MS for titanium carbide and vanadium carbide particles in steels was improved by the application of sulfur-free surfactant which can avoid the mass spectral interferences from sulfur. Finally, the chemical reaction between the sulfides and copper ion extracted from steel sheets was suppressed by using chelate reagents including polyamine structure. Based on the above approaches, a rapid characterization method to determine the size distribution of various nanometer scaled precipitates with different chemical composition in steel has been developed by using SPEED and AF4-ICP-MS.

（英訳）※和文要旨の場合(300 words)
If the abstract is written in Japanese, needed to translate into English.(300 words)