Characterization of Precipitates in Petroleum Steels by Using Precession Electron Diffraction Technique

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Background incl. Aims
Steel has wide variety of grades and properties and not every type of steel applicable for all applications. Due to that reason, correct type of steel needs to be selected for a specific application. For example, for the petroleum and natural gas pipeline applications where pressurized fluid is transported for long distances, American Petroleum Institute (API) X70, X80 or X100 steels are used due to their properties such as high strength, fracture toughness, weldability, corrosion resistance and deformability. API steels are ferritic steels and to obtain the desired properties like high strength, this type of steels is micro alloyed with elements such as Nb, Ti and V with precipitation hardening. Properties of API steels, are modified by the thermo-mechanical rolling by tailoring the microstructure, in addition to the micro alloying. During production and rolling processes several precipitates with different compositions can be formed. Therefore, it is important to determine which precipitate is present in the produced steels, because mechanical properties of material may change with the type of the precipitate. This study aims the characterization of precipitate particle found in the petroleum steel by both conventional and novel precession electron diffraction methods in order to confirm which phase is formed during production.

Methods
In this study, thermomechanically rolled API-X70 steel sample was used. Samples are characterized by conventional TEM and STEM imaging techniques and in addition Precession Electron Diffraction (PED) technique is applied to characterize present phases as well as orientation relationship between the different phases. TEM sample was prepared with conventional mechanical thinning followed by an Ar-ion beam milling. Electron transparent sample is examined by using 200 keV field emission TEM (JEOL-JEM2100F) equipped with STEM high angle annular dark field (HAADF) detector (Fischione-Model 3000) and energy dispersive X-ray (EDX) spectrometer (JEOL-JED2300T). The TEM phase and orientation mapping results are obtained with nano sized probe (at NBD alpha 5 and 0.5 nm spot size) that is scanned in a user specified area over the sample up to 0.7° of precession angle and obtained diffraction patterns are analyzed with ASTAR™ V2 via template matching algorithm.

Results
Microstructure of the samples were investigated by the TEM and STEM imaging methods and these images revealed that steel matrix contains high atomic number element containing precipitate particles according to its contrast with respect to matrix. The sizes of the precipitate particles were ranging from 20 to 80 nm. Chemical composition of the precipitates was tried to be analyzed with EDX, however due to the thickness of the sample, x-ray signals were collected both from precipitate and over or underlaying matrix phase. Because of that reason exact chemical composition cannot be differentiated from the elements present in the matrix. In order to determine the type of the precipitate, phase identification was done by using the precession electron diffraction method by using the crystal structure differences between candidate precipitate phases. For this purpose, phase identification was conducted with calculation and template matching of diffraction patterns of all the possible precipitate phases with experimental diffraction patterns. With this method, phase identification with high reliability was conducted with the additional X-ray spectrum obtained from the precipitate particle.

Conclusions
In this study, precipitate particle present in the API-X70 steel was successfully identified by using different TEM/STEM techniques, which cannot be identified by the EBSD due to the size of the precipitates. For the identification of the both X-ray spectrum as well as phase maps, obtained by the precession electron diffraction method, were used as a complimentary technique to each other in order to eliminate limitations of these methods.

Keywords:

PED, Steel, Precipitate, Phase identification