MLF Experimental Report	提出日 Date of Report June 10, 2016
課題番号 Project No.2015A0153 実験課題名 Title of experiment *In situ* evaluation of dislocation density/structure and texture during thermos-mechanically controlled processing for steels 実験責任者名 Name of principal investigator Yo TOMOTA 所属 Affiliation: National Institute for Materials Science	装置責任者 Name of responsible person K. Aizawa and S. Harjo 装置名 Name of Instrument/（BL No.） TAKUMI (BL19) 実施日 Date of Experiment Nov. 6-8, 2015 (2 days) May 26-28, 2016 (2 days)

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

- Fe-1.5Mn-1.5Si-0.2C (mass %) alloy, Solid
- Fe-2Mn-0.2C (mass%) alloy, Solid
- Fe-0.8C (mass %) alloy, Solid
- Fe-3Cr-1.5Ni-0.4C (mass%) alloy, Solid

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

The changes in microstructure including phase fraction and dislocation density with plastic deformation and/or annealing were measured *in situ* at elevated temperatures using neutron diffraction for four steels. In the first beam time (2 days in Nov. 2015), using a dilatometer, ferrite-austenite transformation were attempted to study but the attachment of thermo-couples to a specimen was not good and hence re-examination was performed at the second beam time (2 days in May 2016). Although the thermo-mechanically controlled processing planned in the proposal was mostly postponed to 2016A because of little time to change to a loading jig, very exciting results were obtained: the three outstanding results were highlighted below.

(1) The austenite-ferrite transformation behavior during heating and cooling in Mn-Si-C steels were successfully monitored at the 2nd beam time. The volume fractions of the constituents showed good agreements with those estimated from dilatometry. This result reveals how neutron diffraction is useful to study steel making process because other *in situ* monitoring methods like SEM/EBSD and X-ray diffraction were of no use because the chemistry near specimen surface changes due to Mn and C evaporation upon heating. Figure 1 presents the comparison of austenite volume fraction during heating determined by dilatometry, neutron, X-ray and SEM/EBSD to demonstrate that neuron diffraction is the most powerful method. Different from dilatometry, neutron diffraction provides not only the changes in volume fractions of the constituents but also lattice constant and microstructural parameters like dislocation density and texture. This result will be published combining the experimental results obtained by several related methods in near future.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

(2) Another important phenomenon for steel production is pearlite transformation (austenite to ferrite and cementite). We already found the diffraction line broadening in patented and cementite-spheroidized pearlite steels but not in recrystallized ferrite-cementite steels at room temperature. In this study, the line broadening was firstly found to occur with pearlite transformation, stemming from the misfit strains at the ferrite-cementite interface (see Fig. 2). After finishing the transformation, temperature was elevated to a higher holding temperature where cementite plate changed to spherical resulting in a decrease of line broadening. This new finding was reported at the ISIJ Spring meeting in 2016.

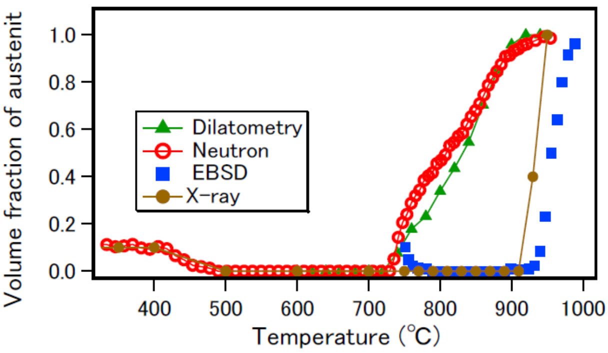

Fig. 1 Change in austenite volume fraction with annealing for a TRIP steel. The retained austenite was dissolved around 450℃ and then reversion from ferrite to austenite was found at 730℃ showing good agreements between neutron diffraction and dilatometry. However, the reverse transformation temperatures determined by *in situ* X-ray and SEM/EBSD were too high because of Mn evaporation from the specimen surface resulting in incorrect data.

(3) One more interesting result obtained is the change in diffraction intensity with recrystallization after reverse transformation from tempered martensite to austenite in a Cr carbide containing steel. Upon heating, first the transformation from ferrite (tempered martensite) to austenite took place where dislocation density was extremely high and then re-crystallization was found to occur accompanying the decrease in dislocation density that was determined using the CMWP fitting method. This result, so-called second recrystallization of transformed austenite has been predicted from several ex-situ measurements and microstructure observation in industry, then showed such a change using *in situ* SEM/EBSD observations by the present workers (2015) and finally verified as *in situ* global bulk measurement with neutron diffraction. The result will be published within in a couple of years.

The unfinished experiments written in the proposal will be continuously performed in 2016A 0127 (accepted).

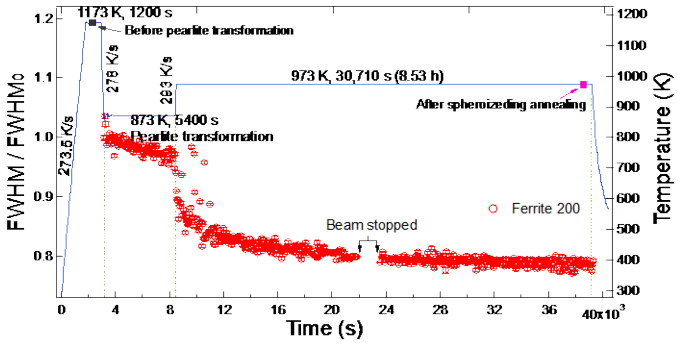

Fig. 1 Change in Full-Width at Half Maximum (FWHM) during pearlite transformation followed by cementite spheroidizing.

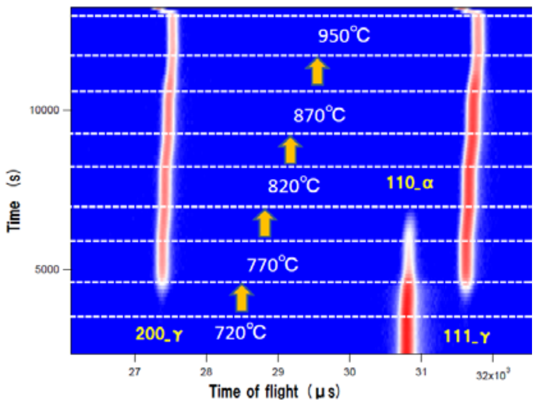

Fig. 1 Austenite (γ) transformation monitoring on heating of a tempered martensite (α) specimen with in situ neutron diffraction in vacuum.