MLF Experimental Report

課題番号 Project No. 2010B0019	提出日 Date of Report March 8, 2011
実験課題名 Title of experiment In situ neutron diffraction and dilatometric measurements for tempering behavior of martensite steels	装置責任者 Name of responsible person Kazuya Aizawa
実験責任者名 Name of principal investigator: Yo Tomota	装置名 Name of Instrument/(BL No.) Engineering materials diffractometer (BL-19)
所属 Affiliation: Ibaraki University	実施日 Date of Experiment Dec.10 – Dec.14, 2010

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.

The chemical compositions of high nitrogen martensitic steel are 0.1C–0.6N–16Cr–1.0Mo–0.6V (mass %). The steel was melted in a nitrogen atmosphere and an ingot was forged to a plate. Small specimens cut from the plate were homogenized and solution treated at 1423K for 1.8ks followed by quenching into water. The microstructure of the as-quenched specimen consists of martensite and the retained austenite. Another steel with chemical compositions of 0.79C–1.51Si–1.98Mn–0.98Cr–0.24Mo–1.06Al–1.58Co (mass%) was also used for reference. For this steel, the microstructure was adjusted to be bainitic ferrite with the retained austenite. The dissolution of austenite with tempering was common objective to be made clear.

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 size of a specimen used for simultaneous measurement of dilatometry and neutron diffraction was 5 by 5 by 30 mm (rod). A dilatometer was set at the Engineering materials diffractometer (TAKUMI) in such a way that the longitudinal direction of the rod specimen was 45 degrees with respect to the incident neutron beam. Hence, the data along the longitudinal and transverse directions were obtained at the north and south detector bank. In order to examine microstructure change with tempering, a specimen was heated step by step like in Fig. 1. A little different heating schedule was also examined. In case of bainitic steel, the specimen heated up to 900℃ were cooled down to 300℃ and kept there to observe bainitic transformation..

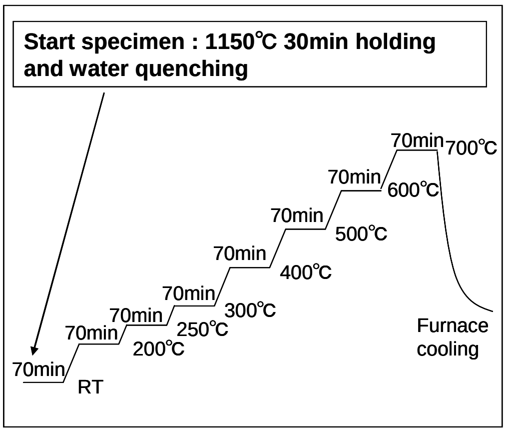

Fig. 1 Heating schedule for tempering study for the high nitrogen bearing steel.

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

Examples of diffraction profiles obtained are presented in Fig. 2. As seen, the starting microstructure is composed of martensite and austenite. At 600℃, all the peaks shift toward right hand side because of thermal expansion. The FWHM was decreased at 600℃ and became much smaller after heating indicating the tempering of martensite. The precipitation of Cr nitride was found at 600℃. To be noted here is the decomposition of the retained austenite as is summarized in Fig. 3. In case of Fe-C martensite, the retained austenite is decomposed to ferrite and cementite around 280℃. However, the present high nitrogen bearing austenite is stable even heating at 400℃ and starts to decompose around 500℃.. The decrease in FWHM for martensite peaks suggest that transformation induced dislocations in the quenched martensite are more difficult to be annihilated than in the conventional carbon martensite. This high nitrogen steel is characterized by showing the secondary hardening by tempering as is observed in Fig. 4, so that high strength can be achieved at higher temperature like 500℃. The secondary hardening is caused by the precipitation of nano-sized Cr nitride, that was confirmed by means of small angle neutron scattering study. But the present data claim the high density of dislocations at higher temperature contributes also to suppress softening with tempering.

The change in specimen length informs us microstructure evolution, for example, expansion corresponding to the austenite decomposition, but more comprehensive information can be obtained from neutron diffraction from a bulk specimen.

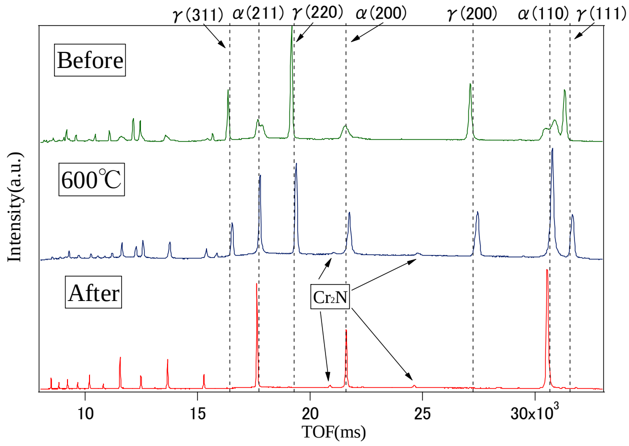

Fig. 2 Example of neutron diffraction profiles obtained

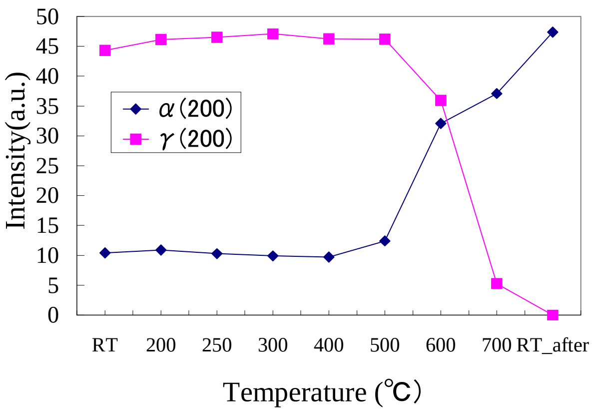

Fig. 3 Diffraction intensities as a function of temperature for 200 martensite (or ferrite) and 200 austenite.

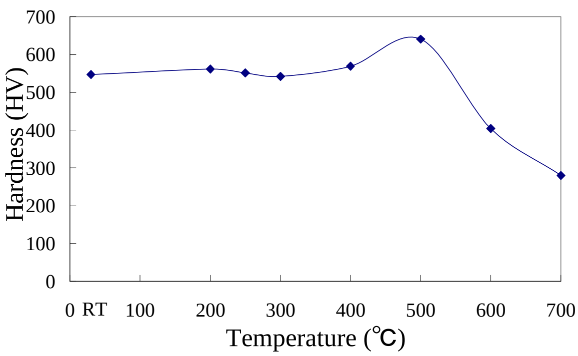

Fig. 4 Hardness change with tempering (1h at each temperature).