MLF Experimental Report	提出日 Date of Report 01/07/2011
課題番号 Project No. 2009B0042 実験課題名 Title of experiment Local Crystal Rotation and Misfit Strain after High Temperature Creep in a Single-Crystal Nickel-Base Superalloy 実験責任者名 Name of principal investigator Yoshinori Shiota 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of responsible person Kazuya Aizawa 装置名 Name of Instrument/（BL No.） TAKUMI/(BL 19) 実施日 Date of Experiment 09-10/05/2010

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.

Name of sample : Single-crystal nickel-base superalloy (TMS-75)

Chemical formula : γ (Ni) matrix and γ' (Ni3Al) coherent precipitate phase

Compositions (atomic%) : Ni 63.0, Al 13.7, Co 12.6, Cr 3.6, Ta 2.1, W 2.0, Re 1.66, Mo 1.3, Hf 0.03

Physical form : Tensile bar with uniform deformation part of 4mm(R) x 20mm(L)

State of sample : After high temperature creep test that is 200hr-creep time deformation with 137MPa at 1100degrees C.

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.

[Experimental method]

The relative sample of single-crystal nickel-base superalloy was set up horizontally in penta-axial goniometer on the TAKUMI sample stage. The angle between scattering direction and incident neutron beam was 45 degree. Because the sample was single crystal as γ and γ' phases with same crystal orientation, the lattice planes (h00), (0k0) and (00l) of two phases were measured at same time. Neutron gauge volume was selected as 1x1x1mm^3 at south detector bank by using small radial collimator. The scanning steps were 13 points along axial direction in the sample. Measured neutron diffraction data was converted into two forms. One form was profile data (2D: TOF and Intensity) from total channel. Another one was pixel data (3D: TOF, Intensity and Pixel) from channel by channel.

[Results]

Different with previous experiments based on the average information on the entire sample, the local crystal rotation and misfit strain measurement was evaluated in this experiment by high-spacial resolution neutron diffraction to clarify the mechanism of the creep damage evolution.

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

Considering that the high-temperature creep deformation may result in the local crystal rotation of γ/γ' phases at one domain position, the local crystal rotation of a super-alloy specimen after 200hours creep deformed at 1373K was measured. Fig. 1(a) explained how the pixel information of time-of-flight neutron diffraction spectra was employed to describe the local orientation of γ/γ' phases of super-alloy, while Fig. 1(b) summarized all the local crystal orientations observed from different pixels on detector. Here, a small change in the crystallographic orientation of monocrystalline was found by seeing the diffraction information separately by each pixel of the detector. The initial crystal orientation was straight line (χ is zero) before creep deformation, while the local crystal orientation rotation rotated gradually in proportion to the creep deformation, and the microcracks initiated in the deformation region before rupture. The local creep deformation was caused by the accumulation of slip deformation. Therefore, the distributions of local crystal orientations of the γ and γ' phases by neutron diffraction can then be measured to evaluate damage process in a non-destructive method, and to provide quantitative information for predicting the lifetime of Ni-base superalloy parts. At the same time, the misfit strain ($=2(a^{\gamma'}-a^{\gamma})/(a^{\gamma'}+a^{\gamma})$) between the γ matrix and the γ' phase was evaluated as a microstructural deformation parameter, related to the local crystal rotation. However, only one sample was measured after creep deformation, and the relation between the local crystal rotation and the misfit was not clear. Therefore, in-situ evaluation of high temperature creep deformation behavior is necessary to clarify the mechanism of the creep damage evolution. At TAKUMI/J-PARC, the device performance including optional environment and the neutron beam intensity have enough ability for this in-situ measurement, and we are planning next experiment.

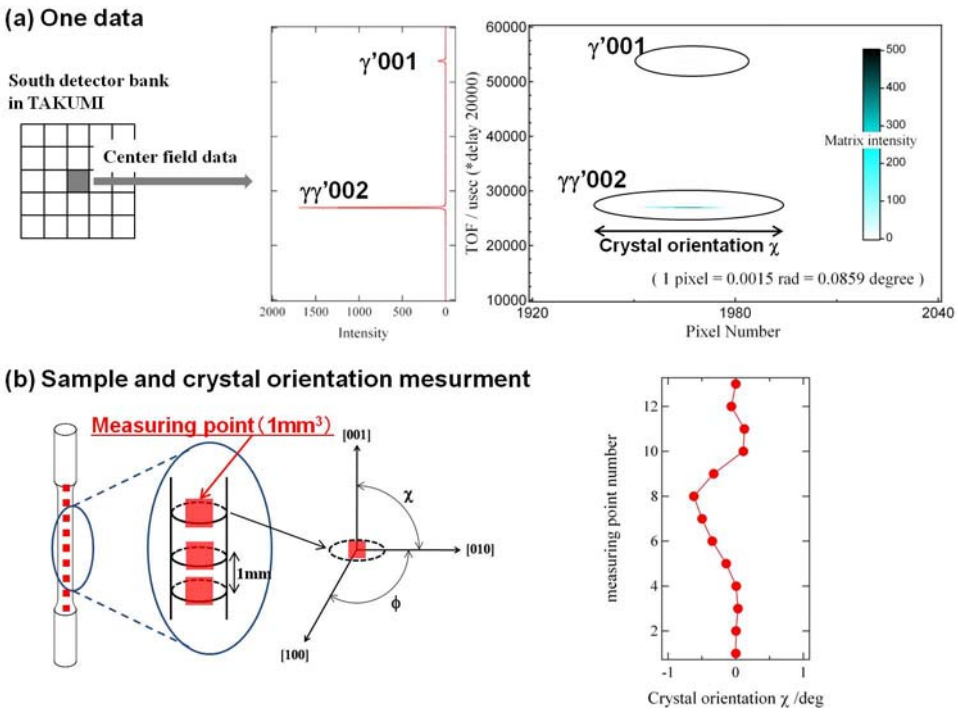

Figure 1 Experimental results about local crystal rotation of Ni-base superalloy after 1100degreeC-200hr creep test obtained from TAKUMI neutron diffraction.