CROSS TOKAI Experimental Report J-PARC	承認日 Date of Approval 2016/12/4 承認者 Approver Jun-ichi SUZUKI 提出日 Date of Report 2016/12/4
課題番号 Project No. 2015A0026 実験課題名 Title of experiment SANS study of the kinetics of nanoparticles in oxide dispersion strengthened (ODS) steels 実験責任者名 Name of principal investigator Hiroaki MAMIYA 所属 Affiliation National Institute for Materials Science	装置責任者 Name of Instrument scientist Jun-ichi SUZUKI 装置名 Name of Instrument/（BL No.） TAIKAN/BL-15 実施日 Date of Experiment 22nd Feb. to 23rd Feb.

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.

Sample 1: 17.8Cr-13.35Ni-2.2Mo-0.09Mn-0.86Si-0.02C-Fe(Bal.)
Sample 2: 17.8Cr-13.35Ni-2.2Mo-0.09Mn-0.86Si-0.02C-Fe(Bal.)
Sample 3: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 4: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 7: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 8: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 11: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 12: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 3_700, Sample 3_800, Sample 3_900, Sample 3_1000: 13Cr-30Mn-0.5N-0.35Y2O3-Fe(Bal.)
Sample 16: 16Cr-15Co-5Ti-3Mo-2.5Al-1.5W-Ni(Bal.)
Sample 17: 16Cr-15Co-5Ti-3Mo-2.5Al-1.5W-Ni(Bal.)

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 samples were measured by the small angle neutron scattering (SANS) instrument TAIKAN.

The material was manufactured from elemental powders of Fe, Cr, Mn and Y_2O_3 by mechanical alloying under the nitrogen atmosphere. After 40h of milling powder was sintered in four conditions 5 min or 15 min and 950ºC or 1000ºC. To investigate thermal stability one sample, sintered in 1000 ºC for 5 min, was annealed in four temperatures, 700, 800 900 and 1000 ºC.

For all of them, the direct beam and scattering from glassy carbon were measured. The measurements were made for 90000 or 80000 pulses, depending on the sample. The measurements were held in the ambient temperature with nonpolarized neutron. The q range was 0.005 $Å^{-1} < q < 0.2$ $Å^{-1}$. The measured data was corrected using the results of the glassy carbon, thickness and analyzed by Irena software.

In the Fig. 1 are presented results of SANS measurement of samples sintered in four various conditions, which show influence of sintering conditions on the nanoparticles size distribution.

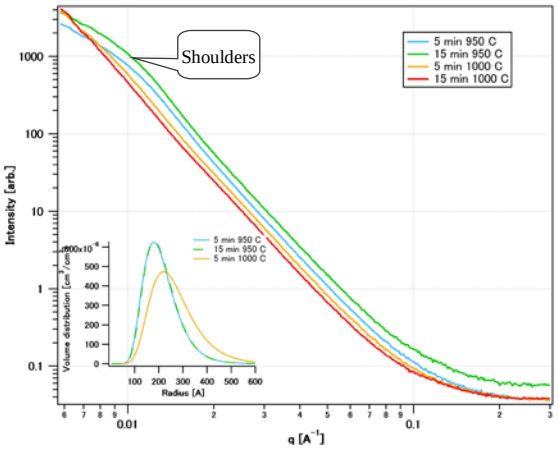

Figure 1 SANS, samples sintered in four conditions

For samples sintered in 950ºC, we can observe clear shoulders, in the q < 0.016 Å^{-1} region. There is also observed shoulder for sample sintered in for 5 min in 1000 ºC, but it is less visible because of shifting to higher q value and lower intensity. In case of sample sintered for 15 min in 1000 ºC there is not visible any shoulder. The analysis of the results by Irena shows that samples sintered in 950 ºC are characterized by similar volume size distribution of nanoparticles with size around 230 Å. In case of the other sample, the curve shows wider distribution of of slightly bigger nanoparticles.

The results show the main factor controlling their size is temperature. However holding time is not without significance. The yellow curve of samples sintered for 15 min in 1000 ºC continuously rises to samll q value, which suggests, there are bigger nanoparticles, which could not be detected in the q range.

The SANS results were confimed by TEM observation. However the pictures show existence of nanoparticle with very various size and shape. We could observe nanoparticle smaller than 100 Å.

None the less SANS as a bulk method indicated the majority. The EDS analysis shows that nanoparticles with size around 400 Å are manganese oxides. It is negative phenomena, which causes depletion of matrix and can lead to brittleness of the matieral.

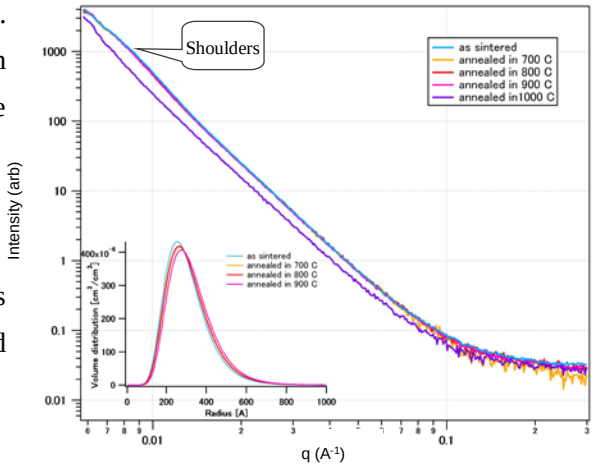

Figure 2 SANS, annealed samples

SANS results of annealed samples show shoulders in 0.006 Å^{-1} < q < 0.012 Å^{-1} region. We can observe slight shoulder shifting to lower q value, with annealing temperature, up to 900 ºC. However it is not significant. In case of sample sintered in 1000 ºC, we could not observe any shoulder. Possibly nanoparticles are not any more thermally stable, which caused their growth so much we cannot observe them in the q range (Figure 2). It express the manganese oxides are stable up to 900ºC.

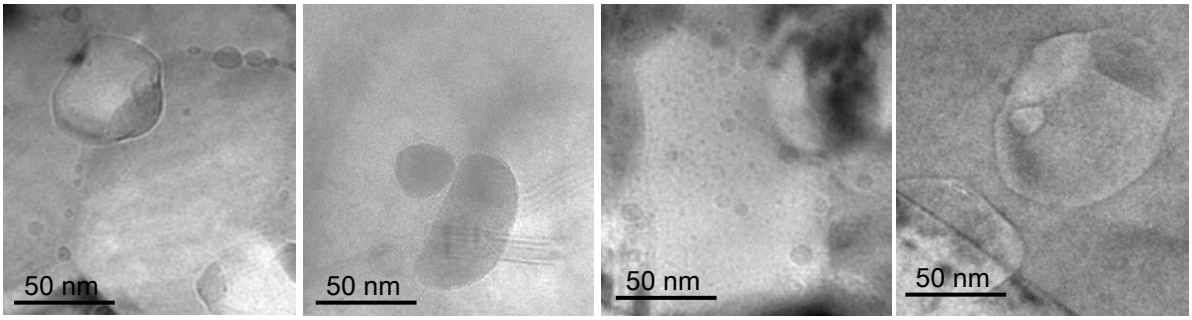

Figure 3 TEM, nanoparticles of samples sintered in various conditions