MLF Experimental Report	提出日 Date of report 2013/05/20
実験課題番号 Project No. 2012P0102 実験課題名 Title of experiment Strength of Materials and Related Engineering 実験責任者名 Name of principal investigator Y. Tomota 所属 Affiliation Ibaraki University	装置責任者 Name of responsible person K. Aizawa 装置名 Name of Instrument/（BL No.） TAKUMI (BL19) 利用期間 Dates of experiments 2012/04/24 – 2013/03/16 （16 times）

1．研究成果概要（試料の名称、組成、物理的・化学的性状を明記するとともに、実験方法、利用の結果得られた主なデータ、考察、結論、図表等を記述してください。

Outline of experimental results (experimental method and results should be reported including sample information such as composition, physical and/or chemical characteristics.

Overall outline of the experimental results in the project

This project includes three topics, *i.e.*, (1) Mg based synchronized LPSO alloys (Topic A), (2) Commercial superconductor composite materials (Topic B), and (3) Advanced steels (Topic C). The common research issues are analyses and evaluation of crystal structure, texture, constituent phases, microstructure (phase volume fraction etc.), macroscopic (Type 1) stress, phase and intergranular (Type 2) stress, densities of lattice defects like dislocation, crystallite size and so on by employing neutron diffraction measurements. Mesoscopic scaled information, for example, the size and distribution of nano-precipitates, could be evaluated by neutron small angle scattering measurement. It is, however, the application of such neutron techniques to industrial materials has just started, so that many problems to hinder evaluation of such parameters with sufficient reliability have to be overcome. More appropriate methods for measurements and data analyses for accurate determination of microstructural parameters, in situ measurement during material processing as well as mechanical response of the above engineering materials were investigated. High impacts of the present research to industry can be expected. The main results of the three topics are as follows.

Topic A： Crystal structure and mechanical properties of Mg based synchronized LPSO alloys

This research was carried out through the KAKENHI, innovative research project of Material Science of Synchronized LPSO alloys – Innovative developments towards the next generation light structural materials - (H23 – H27). This project is composed of (1) Crystal structure science, (2) Microstructural evolution mechanism, and (3) Mechanical properties and elucidation of new strengthening mechanism. In order to bridge these issues, the mission of this group is to perform the precise atomic structure analysis to make clear the mechanism of characteristic mechanical response of the LPSO alloys using neutron beam. Crystal structure analysis by neutron diffraction, *in situ* neutron diffraction during deformation and small angle neutron scattering experiments were carried out for the LPSO alloys which were made by another group of the above KAKENHI project.

１．研究成果概要（つづき） Outline of experimental results (continued).

The obtained results by neutron experiments will be discussed by comparing with theoretical computation using the first principle theory, experiments using synchrotron X-ray etc. , aiming at making clear the real characters of LPSO structure.

Because Mg-based synchronized LPSO structure has more than five repetitions along the c-axis, it is required to obtain higher indexed peaks in order to determine its precise structure. It is necessary to obtain diffraction peaks up to higher index in order to determine precise crystal structure. Hence, it is important to develop the wide range wavelength measurement for Takumi. The evaluation of phase stress, dislocation density, crystallite size etc. is indispensable to obtain the deep understanding of mechanical response and strengthening mechanism of LPSO alloys. For samples prepared from extruded rod, uni-directionally solidified ingot and others, the development of a small scaled machine to employ compact tensile specimens are also needed. In H24 fiscal year, a compact deformation stage with a furnace was designed and installed through the above KAKENHI project.

It is also important to develop the software to determine microstructural parameters like dislocation density and crystallite size, so that we invited Prof. Ungár of Eötvös University, Hungary, who is one of the authorities of line profile analysis in order to introduce their software to TAKUMI.

In addition, the combinational data acquisition system for the conventional and the newly developed compact loading machines should be realized. In the H24 fiscal year, such a data acquisition system combined with the conventional tester and the data acquisition system has been finished. A two-axial rotation equipment with a Gandolfi camera was installed in order to avoid the influence of texture to neutron diffraction pattern.

In this fiscal year, experiments were carried out five times for in situ neutron diffraction during deformation and ex-situ neutron diffraction using the Gandolfi camera.

(1) 2012_5/12-5/14 : LPSO alloys (Uni-directional solidification samples: Mg_{85}Zn6Y_9, cast poly-crystalline samples: $Mg_{97}Zn_1Y_2$ and $Mg_{85}Zn_6Y_9$, and excluded samples: $Mg_{97}Zn_1Y_2$ and $Mg_{89}Zn_4Y_7$). Neutron diffraction pattern measurement.

(2) 2012_6/13-6/14: LPSO alloys, In-situ neutron diffraction during tensile deformation.

(3) 2012_12/13-12/14: LPSO alloys, In-situ neutron diffraction during compression deformation.

(4) 2013_02/26-2/28: LPSO alloys, Pure zinc, Rocking curve measurement during compression.

(5) 2013_04/08-04/09: LPSO alloys, In-situ neutron diffraction during tensile deformation, Neutron diffraction pattern measurement on the Gandolfi goniometer.

It is planned as the next step that in situ neutron diffraction will be performed during heat treatments including solidification, solid phase transformation at elevated temperatures. The newly developed tensile tester with a compact furnace will be used for diffraction measurements as well as small angle scattering at BL15, TAIKAN at elevated temperatures.

In fiscal 2012, Mg based synchronized LPSO alloys (uni-directionally solidified $Mg_{85}Zn_6Y_9$ (18R LPSO single phase sample), as cast $Mg_{85}Zn_6Y_9$ (18R LPSO single phase), extruded $Mg_{89}Zn_4Y_7$ (18R LPSO 相、α-Mg two phase), cast $Mg_{97}Zn_1Y_2$ (18R LPSO phase, α-Mg two-phase samples). Extruded $Mg_{97}Zn_1Y_2$ (18R LPSO phase, α-Mg 2 phase samples) were examined. As results, it has been found that the crystal structure of 18R LPSO determined by neutron diffraction shows a good coincidence with that proposed by TEM

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１．研究成果概要（つづき） Outline of experimental results (continued).

observations, *i.e*., $P3_212$, a=1.11, c=4.69 nm). It is also found that the 18R LPSO single phase alloy exhibits anisotropic mechanical response with respect to the a- and c-axis. In two-phase alloys, the role of strengthening is attributed to the 18R LPSO phase.

In 2013, further crystal structure analysis will be progressed using neutron diffraction measurements with two-axial rotation by the Gandolfi camera and the strengthening mechanism by kink band deformation will be investigated using in situ neutron diffraction during compressive deformation and small angle neutron diffraction. To elucidate the formation mechanisms of Type I LPSO alloy, Mg-Zn-Y, and type II Mg based synchronized LPSO alloy, Mg-Zn-Gd and Mg-Al-Gd, will be examined using high temperature neutron diffraction and small angle scattering.

Topic B: Influence of stress and/or strain on commercial super conductive composite materials

To make clear the influence of stress and/or strain on superconductive properties in commercially available super conductive composite materials, the mechanism of strain generation and its influence on superconductive properties have been studied. In 2012, neutron diffraction measurements at room and elevated temperatures were performed and the adjustment of a tensile tester at cryogenic temperatures was made. A part of the experiment was performed as general proposals and their flexible beam time arrangement with this project was very effective.

Tensile strain evaluation of Nb_3Sn Rutherford cables (Tohoku University: April 26-27, 2012 and March 20-21, 2013)

Measurements of the residual elastic strains at room temperature and internal strains under external tensile load were performed through neutron diffraction for the 16 wire-wired Rutherford cable made of Cu-Nb strengthened Nb_3Sn wire（φ0.8mm） fabricated by the Nb rod method. The two kinds of specimens were prepared; one was pre-bent using jig with 65 mm in diameter after wire-twisting (Prebent φ65) and the other was not bent (w/o Prebent). The value for d_0 was obtained by the filaments extracted from the same batch wires. It was found that the residual compressive strain of approximately -0.2% in Nb_3Sn for the c/o Prebent sample was relaxed to -0.06% by pre-bending. The strains under tension is now in analyzing.

High temperature measurement of internal strain of Nb_3Sn and Nb_3Al wires (Appl. Sci. Research Institute and Daido Univ., Nov.23-23. 2012)

The temperature T_0 for the onset of thermal stress generation was determined in order to estimate residual thermal strains for Nb_3Sn and Nb_3Alwire. The obtained results were T_0=421K and 380K for Nb_3Sn and Nb_3Alwire, respectively. In addition to the evaluation of thermal strains, it was revealed thermal strain changed from compressive to tensile above relevant T_0 temperature. Judging from the experimental results and theoretical calculations, it is concluded to be caused by larger thermal expansion rate of pure copper compared with that of Nb. Hence, it is possible to estimate thermal stains near 4.2 K using this calculation method.

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Internal strain measurements of the superconducting wires soldered on the spring (RT) (KEK,NIMS：Dec. 1-2,2012)

As a method to measure the critical current density of a super-conducting wire under the external magnetic field with applied condition of either tensile or compressive strains along the wire axial direction, to solder the super-conductor wires to a jig has been used. The problems of this method include that thermal stresses generated due to the difference of thermal expansion rate between the wire and the jig. Therefore, the residual strains along the all directions planned were measured for the soldered state of the wire on the jig. The strains determined from the diffraction peak shifts for Nb_3Sn filaments were, -0.086 % in the axial direction and 0.009 % in the transverse direction for the wire sample, -0.149 % in the axial direction, 0.008 % in the transverse, and 0.041 % in the depth direction for the spring sample. From these results, it is concluded that the compressive residual strain increases along the axial direction by soldering. On the contrary, the changes in strains along transverse directions were found much smaller.

Tensile strain effects of Nb_3Al wires at RT（KEK: April 24-25, 2012）

Tensile tests were performed for Cu-plated Ta-matrix RHQ-Nb_3Al wires at room temperature. As results, it is revealed that approximately 0.03 – 0.05 % tensile strains were remained in both of the axial and radial directions. The linear relationship between the macroscopic strain measured by an extensometer and mean lattice strain measured by neutron diffraction was found and its slope was about 0.76 in the axial direction and 0.78 in the radial, both of which are lower than unity. In the next step, low temperature tensile tests and analysis on the strain dependence of super-conductor properties will be made.

Related results obtained by another project

Followings were obtained by another project related to this present study.

Development of low temperature load frame（KEK・JAEA：Jan., 13-14, Feb. 9-10）

A new tensile tester for cryogenic use was developed through the budgets of KEK and JAEA. Trail experiments to adjust the experimental set up were carried out and tensile tests at 8 K were finished for a stainless steel and for a Nb_3Al wire.

Internal strain states in ITER-CICC Nb_3Sn conductors（JAEA：June 17-20, 201）

Residual strain measurements were performed for a CICC conductor of ITER-CS（center solenoid）. A bundle of the CICC conductor wires (32.6 mm in diameter) was inserted into a 40 mm SUS steel composite. In the SULTAN experiment, the transition temperature to super conductivity, Tsc, has been measured by loading large power current under a magnetic field. The internal strain distribution for a specimen revealed deterioration of Tcs by this experiment were measured. As results, residual strain was found larger in the region subjected to lower electromagnetic power than in the region higher power. Then, it could be concluded that the wire displaced toward the direction of electronic-magnetic direction resulting in space in the opposite side and that the twisted wires were bent strongly to be deteriorated. Such a model can newly be proposed.

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Topic C: Development of advanced steels using in situ neutron diffraction

In situ neutron diffraction measurements were performed four times focusing on tension(-compression) deformation, heat treatments, and thermo-mechanically controlled processing. The experiments include,

5/10-5/11: Fe-Si alloy, Fe-C alloy (pearlite)

11/5-11/: Fe-Mn-Si-C (TRIP-DP, SCM490) alloy, Fe-Si alloy, Fe-C alloy, Tensile deformation at RT.

12/17-12/18: Fe-Mn-Si-C alloy, electrodeposited Fe, Fe-Ni alloy, Heat treatment using a dilatometer.

2/16-2/17: Fe-Mn-Si-C alloy; Fe-Mn-C alloy, Cyclic heating and TMCP simulation: in situ neutron diffraction measurements during room temperature deformation were carried out for three kinds of steels.

In case of Fe-Si alloy, the increase of work hardening with increasing of Si concentration were investigated and it was found that the Si addition suppress the texture change and increase of dislocation density. Such changes in dislocation substructure is believed to cause the increasing of intergranular stress. The results are consistent quantitatively with the TEM observations.

In lamellar pearlite (ferrite-cementite) steel, stress partitioning between ferrite and cementite were reconfirmed. In addition, the peak broadening in cementite peaks at the early stage of plastic deformation was clearly observed. It is needed to take into consideration the inhomogeneity effect for determination of dislocation density and crystallite size using line profile analysis. Such microstructure analysis not only for the ferrite matrix but also cementite is future work.

In TRIP/DP steels for automobile use, it has become possible to separate martensite (bct) peaks from ferrite (bcc) ones, so that stress partitioning between the two phases could be determined.

Using a dilatometer, grain growth behavior in a ultra-fine grained electro-deposited iron with heating was tracked by in situ neutron diffraction. Grain boundaries were suspected to be pinned by hydrogen pores and then, the dispersion of hydrogen induced abnormal grain growth (so called 2^{nd} recrystallization) accompanying texture change. These results will be published as an original paper inputting related other experimental results. This must be influential insight for thermal stability of ultra-fine grained materials.

In the experiment on cyclic austenite-ferrite transformation by in situ neutron diffraction, the strong variant selection was revealed to result in texture memory. The reversion in a cold-rolled low carbon steel was also tracked and the results obtained are now in analysis. It is interesting to compare the results of the cold-rolled steel with those of a hot-rolled steel which was already reported to show excellent texture memory.

In TMCP simulation investigation for a low-alloyed steel, ① transformation behavior after tensile or compressive plastic deformation for austenite, ② dynamic ferrite transformation behavior during iso-thermal holding at several temperatures and ③ transformation behavior during continuous cooling after austenite deformation were conducted.

１．研究成果概要（つづき） Outline of experimental results (continued).

To simulate real processing in a factory, it is required to enlarge the cooling speed and increasing the amount of plastic deformation, so that the further experiments are planned to use a themec-master that will be installed to BL19 through Kyoto university (Element Strategic Research for Structural Materials) in the next fiscal year. The details of design of this instrument was discussed in cooperation.

Through above three topics, it is commonly needed to develop the profile analysis in order to determine microstructural parameters like dislocation density, crystallite size and texture. Therefore, the collaborated efforts will be made and some detailed plan has been made as key-issues in the next fiscal year.