(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

MLF Experimental Report	提出日 Date of Report 2017/07/02
課題番号 Project No. 2015A0099 実験課題名 Title of experiment Development of high resolution quantitative resonance absorption and Bragg edge imaging for engineering and metallurgic applications and optimization of single crystal growth 実験責任者名 Name of principal investigator Anton Tremsin 所属 Affiliation University of California at Berkeley, USA	装置責任者 Name of responsible person Kenichi Oikawa 装置名 Name of Instrument/(BL No.) BL10 実施日 Date of Experiment 2016/04/16 21:00 ～ 2016/04/22 09:00 2017/01/18 21:00 ～ 2017/01/21 9:00

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.

1. BaBrCl:Eu gamma scintillator sealed in quartz ampule, 12 mm internal diameter, ~6 cm tall
2. CssLiLaBr6:Ce gamma scintillator sealed in quartz ampule, 12 mm internal diameter, ~6 cm tall
3. Aluminium welds, ~10 cm x 1 cmx5 cm
4. Steel welds, ~2x2x2 cm^3, different filler speed
5. TlBr gamma semiconductor detector material, ~5x5x5 mm^3, 4 pieces
6. NaI gamma scintillator material, ~11 mm internal diameter, ~5 cm tall, sealed in quartz tube
7. CsI gamma scintillator, ~2 cm disk, 10 mm thick

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.

All the samples were measured as planned: the gamma scintillator and semiconductor materials were measured while melted in specially designed and manufactured vacuum furnace installed in neutron beam. The furnace was controlled with a feedback loop for accurate temperature to be maintained in the sample. The sample materials were melted (BBC at 850C and CLLB at 550C) and then slowly grown into single crystal materials for the studies of growth conditions, which can be optimized with the in-situ diagnostics attempted in the present experiments. The results of these measurements on the scintillators are very interesting and are met with great interest by the crystal growth community and are being prepared for the publication in a refereed journal, most likely J. of Crystal Growth and Design.

Neutrons for a broad range of energies (including both thermal and epthermal energies) were registered in each experiment providing the opportunity to study simultaneously the elemental composition of the materials and their crystallographic properties and interface location between the liquid and solid phases. Neutron resonance absorption was used to evaluate the elemental composition of CLLB sample. Concentration of Li is shown in Fig. 1.left, which was reconstructed from the measured spectra while sample was melted to the white

dashed line. The integration time of this image was ~40 minutes, acceptable for the crystal growth processes, although a higher beam intensity will definitely be very useful for quantification of other elements, which is not possible with current neutron statistics acquired in our experiment.

The challenge of energy-resolved imaging with the TOF technique lies in the requirement to detect both position and time for every registered neutron with fluxes exceeding now 10^7 n/cm^2/s. Such high fluxes are necessary for high-resolution imaging studies, where multiple transmission spectra are measured within areas as small as 55x55 μm^2, with as many as >250000 spectra measured at the same time. Analysis of this data allows reconstruction of the entire map of some microstructure parameters with spatial resolution of ~100 μm, all from one measurement. The deficiency of transmission imaging should be pointed out here: only the integral characteristics through the sample thickness are obtained by this technique. However, in some cases that averaged information is sufficient, as demonstrated by the studies of weld cross sections and other samples.

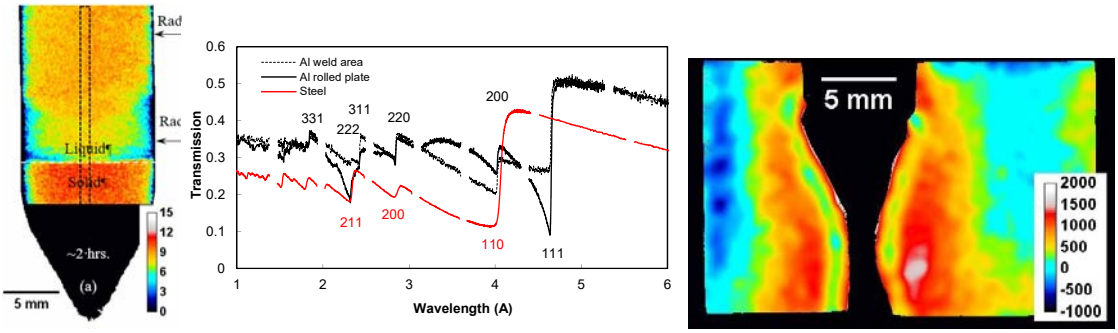

Fig. 1. (Left) Li concentration in Cs2LiLaBr6:Ce sample, measured through energy resolved transmission spectroscopy. **(Center)** Neutron transmission spectra measured at BL10 for various polycrystalline metal samples, Fig. 1.a (~1 cm steel, ~10 cm Al and ~0.5 cm nickel) as well as single-crystal Ni alloy. **(Right)** The map of the residual strain reconstructed from (110) Bragg edge measured within each pixel of our dataset. An accurate fitting of a 5-parameter analytic function allows reconstruction of residual strain with sub-mm spatial resolution, across the entire sample measured in one experiment. The color legend represents the strain values measured in microstrain

The microstructure within relatively thick metal samples was also studied in our experiments through Bragg edges. Typical spectra, measured for steel and Al samples, are shown in Fig. 1. A large difference in the measured spectra of the Al weld sample is observed for the areas within the weld (where friction stir welding produced material with small grains free of strong texture) and within the adjacent rolled plate area, exhibiting a strong texture variation through the sample thickness. Such transmission spectra are obtained in each pixel of our detector in one measurement. The residual strain within a steel weld sample, reconstructed from the measured transmission, is shown in Fig. 1, reveals some microstructure variations formed within the weld area. The location of the filler material is excluded from that image since the filler material was from other metal composition and had different lattice parameter. It demonstrates how the energy resolved imaging helps to visualize the bulk variation of microstructure within the welds and other samples, which can be complementary to many other well established characterization techniques.