

実験報告書様式(一般利用課題・成果公開利用)

(※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。)

	承認日 Date of Approval 2016/12/19 承認者 Approver Takenao Shinohara 提出日 Date of Report 2016/12/19
課題番号 Project No. 2016A0043 実験課題名 Title of experiment Imaging phase-change thermal storage in miscibility gap alloys 実験責任者名 Name of principal investigator Prof. Erich Kisi 所属 Affiliation The University of Newcastle	装置責任者 Name of Instrument scientist Dr Takenao Shinohara 装置名 Name of Instrument/(BL No.) RADEN (BL22) 実施日 Date of Experiment 2016/11/20 – 2016/11/24

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. 40 vol% Bi-Pb alloy and 60 vol% Al in solid form at room temperature. 2. 40 vol% Bi-Pb alloy and 60 vol% C (graphite) in solid form at room temperature. 3. 40 vol% Sn-Pb alloy and 60 vol% Al in solid form at room temperature. 4. 50 vol% Bi-Pb alloy and 50 vol% Al in solid form at room temperature.
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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 experimental arrangement on the beamline is shown in Figure 1. The MCP/Timepix detector (arrowed in yellow) supplied by Dr Tremsin was positioned behind the sample as close as possible. The sample, arrowed in red, was contained inside a silica tube under vacuum. A temperature gradient was created in the sample between a heater at the base and a water cooled cap. Gradients were chosen so that the melting temperature of the included alloy “Bi-Pb” (123 C) or “Sn-Pb” (183 C) was part-way up the detector. The steepness of the temperature gradient was able to be controlled by changing the thermal resistive spacer at the top of the sample between machinable ceramic (3 degrees C/40mm), Pb metal (14 degrees C/40mm) and no resistor (50 degrees C/40mm). Five thermocouples allowed simultaneous measurement of the thermal gradient and the power input to the heater was also recorded so that the overall conductivity can be calculated. Once a stable gradient had been established, energy resolved projection images were recorded. Image recording time was approximately 3h. For sample 1 above, data were recorded with 3 different thermal gradients and with several heater set-points to shift the melt zone to different places within the sample.
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## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

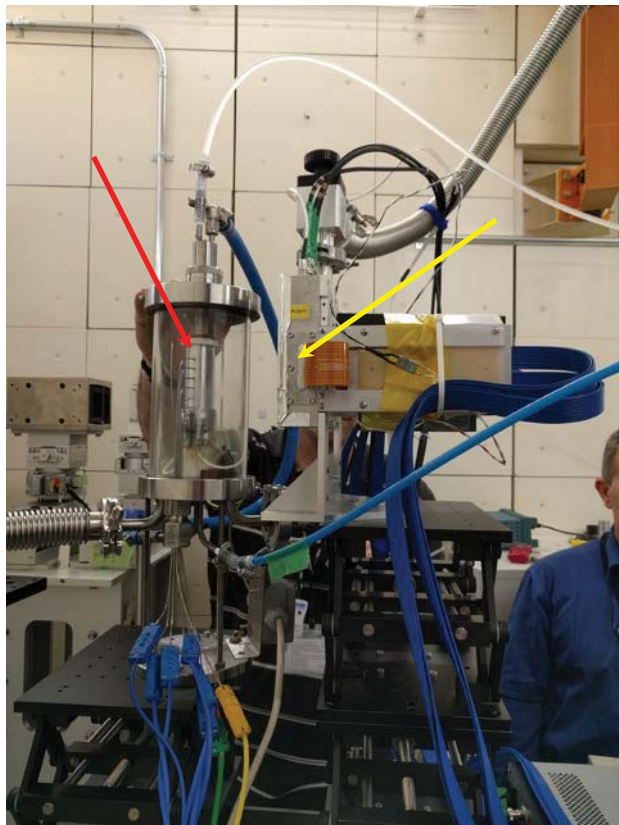


Figure 1 – Experimental arrangement on RADEN.

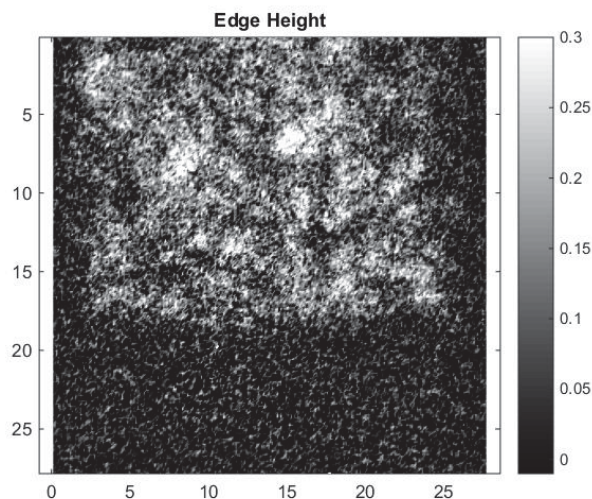


Figure 2 – Bragg Edge image for Sample 1, Pb-spacer

A similar procedure was followed for the other samples to give a good overview of the behaviour of the samples. Open beam images were also recorded before and after the earthquake in case the detector had moved. The earthquake caused only approximately 4h of lost beam time.

Data were initially evaluated using IMAGE-J software to produce Bragg-Edge images. This was later changed to some MatLab scripts that we wrote for this purpose. For all samples, the Pb edge near 5.3814 Å was used for imaging zones where the Pb is solid (edge exists) and where it has melted (edge not visible). An example image is shown in Figure 2. In this image, 2x2 pixel re-binning has been used to improve the statistics. Various types of image processing will be used to further analyze the images and compare with the results of thermal modelling. Of particular interest are the interface sharpness and curvature as a function of the thermal conditions imposed.

In addition to the static images, some images were also recorded during heating to the selected temperatures. Although these data will not be of the same quality as Figure 2, by laterally averaging, it will still be possible to track the average position of the melt zone as a function of time. Additionally, two images were recorded for longer times to attempt the production of Bragg-Edge strain images of the thermal mismatch strains between the phases present in the samples.

Data analysis is well under way and the results are in preparation for publication.