

High-speed *in-situ* measurement of Al metal melting process

Introduction

To capture the moment when materials change, such as during melting, solidification or crystal phase change, by *in-situ* X-ray diffraction measurement, the acquisition time of the X-ray diffraction images at each temperature needs to be as short as possible. 0D and 1D detectors take time to scan the detector and prepare for operation. Conventional 2D detectors also have a problem in that the X-ray shutter needs to be opened and closed between counting and reading the data. The HyPix-3000 hybrid pixel array multi-dimensional detector in 2D mode can acquire X-ray diffraction images without scanning the detector. The HyPix-3000 has two counters inside. Switching between them allows measurement without dead time. These features enable shutterless measurement of 2D X-ray diffraction images, which makes it possible to observe rapid changes in crystalline state.

Measurements and results

2D X-ray diffraction images of an Al plate sample were recorded every 0.5 seconds while rapidly increasing the temperature at 300°C/min. Fig. 1 shows the 2D X-ray diffraction images and schematic views of the crystalline state suggested by their features. Continuous Debye rings from the Al plate were observed at room temperature. This means that the Al crystal had fine grains before heating. During the increase in temperature, the Debye rings became dotted, indicating that grain growth occurred due to heating. When the temperature was increased even further, the Debye rings eventually disappeared due to the melting of Al. It was confirmed that there was a temperature range where the continuity of the Debye rings increased just before melting. This was the moment when the grain boundaries of Al melted and the liquid phase and minute crystal grains of Al coexisted.

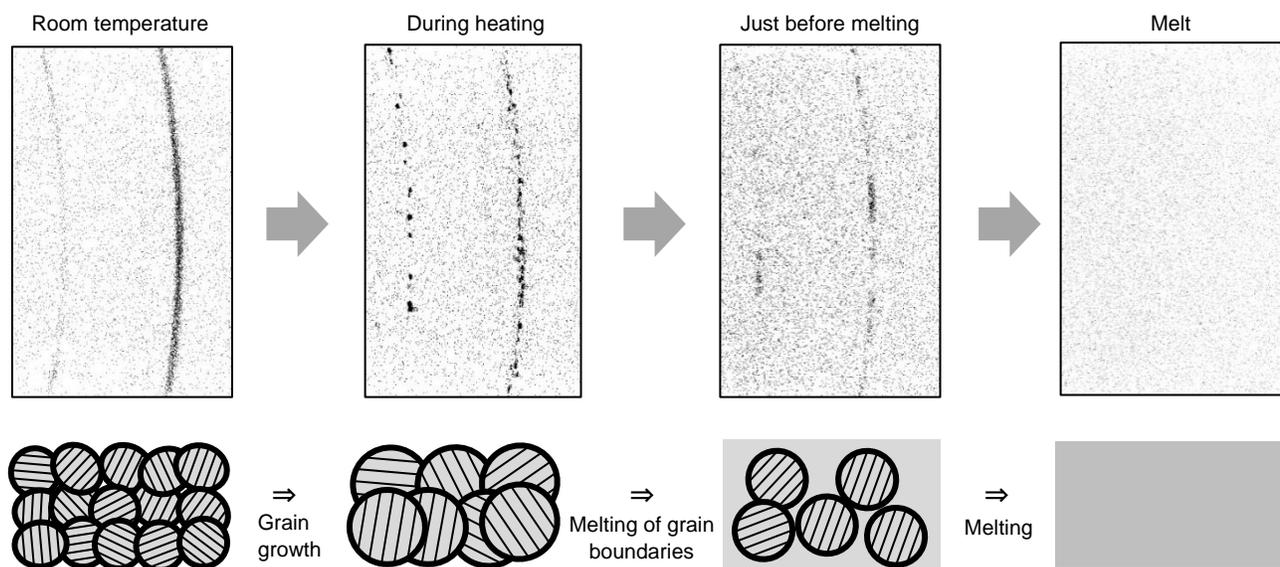


Fig1: 2D X-ray diffraction images observed during high-speed temperature increase *in-situ* measurement of Al metal, and schematic diagrams of changes in crystal grains.

Recommended equipment

- ▶ Automated multipurpose X-ray diffractometer SmartLab
- ▶ Hybrid pixel array multi-dimensional detector HyPix-3000