Available online at www.sciencedirect.com

ScienceDirect

Acta Materialia 81 (2014) 241–247

Acta MATERIALIA

www.elsevier.com/locate/actamat

Retrieval of three-dimensional spatial information from fast in situ two-dimensional synchrotron radiography of solidification microstructure evolution

W.U. Mirihanage [a,*], K.V. Falch [a], I. Snigireva [b], A. Snigirev [b], Y.J. Li [c], L. Arnberg [c], R.H. Mathiesen [a]

[a] Department of Physics, Norwegian University of Science and Technology, N-7491 Trondheim, Norway
[b] European Synchrotron Radiation Facility, 38000 Grenoble, France
[c] Department of Materials Science and Engineering, Norwegian University of Science and Technology, N-7491 Trondheim, Norway

Received 23 June 2014; received in revised form 30 July 2014; accepted 6 August 2014
Available online 7 September 2014

Abstract

In situ synchrotron X-ray radiography of columnar dendritic growth in Al–15 wt.% Cu–9 wt.% Si–0.015 wt.% Sr alloy has been carried out with the temporal and spatial resolutions of 100 ms and 0.65 μm, respectively. Two-dimensional (2-D) projected images have been processed and analysed to retrieve three-dimensional (3-D) spatial information on the growing dendrite network, through X-ray transmission contrast differences that account for solute-composition variations in the liquid and thickness variations in the growing dendrites. The analysis elucidates the way in which gravity-driven natural convection and solute sedimentation can result in solid fractions that deviate markedly from Scheil and Lever-rule-based models, which could alter mushy zone permeability. Furthermore, the ability to render 3-D spatial details may lead to future opportunities to use 2-D radiography in the development and validation of more sophisticated 3-D models of dendritic growth.

Keywords: Solidification; In-situ radiography; Synchrotron radiation

1. Introduction

Fast synchrotron X-radiography [1–4] and X-ray tomography [4–9] have emerged to become well-established tools for studying solidification microstructure formation and evolution in metallic alloy systems in situ. While the nominal spatial resolution offered by these two methods is more or less the same, i.e. ~1 μm unless X-ray optics are employed, their spatiotemporal performance is quite dissimilar. X-ray tomography has the advantage of providing full three-dimensional (3-D) information, but at the cost of approximately two orders of magnitude longer sampling times [5–8]. In addition, tomography does not generally provide on-line vision during experiments since image reconstruction is required. This makes radiography the most convenient choice for studies of solidification fronts dictated by fast transformation kinetics or growth under non-steady-state conditions, while tomography would be the best alternative for studies of somewhat slower phenomena, at low cooling rates or deeper in the mushy zone at later stages of solidification. In a recent synchrotron experiment a polychromatic X-ray beam and a ultra-fast CMOS-based X-ray imaging camera were employed to bring the sampling time down to ~100 ms at 1.5 μm resolution in tomography studies of solidification microstructure evolution in

* Corresponding author. Tel.: +47 45693605.
E-mail address: wajira.mirihanage@ntnu.no (W.U. Mirihanage).

http://dx.doi.org/10.1016/j.actamat.2014.08.016
1359-6454/

Al–20 wt.% Cu [9], focusing on early stages of solidification under a very slow cooling rate of 0.05 K s^{-1}. Yet, since this involves polychromatic X-rays, there are severe trade-offs, e.g. the selection of energy range in a compromise between yielding appreciable image contrast, at the same time avoiding influential sample heating by the X-ray beam; limited potential for constitutional assessment from image contrast; etc. In short, considerable development and a more extensive use of the technique is required before the general output potential of filtered white-beam tomographic imaging can be assessed properly, and it will therefore not be considered any further here at this stage.

For two-dimensional (2-D) projected radiography images to reveal useful details, measures must be taken to prevent the formation of multiple crystal layers stacked along the direction projected by the incident beam. Thus, samples are made as sheets with a thickness limited more or less to the diffusion length of the growth process investigated. In a very rough approximation the sample may be considered as 2-D, yet in more detailed comparisons with theories or microstructure simulation models it will often be necessary to assess the missing 3-D information of a confined volume in order to address important aspects like growth characteristics, mushy zone permeability, solid fractions, dendrite tip morphology and diffusion field, liquid flow, etc. Under certain conditions, however, some information on the third spatial dimension may be retrieved through image processing. If the primary solid phase has relatively high X-ray transparency compared to the initial alloy composition, as would be the case with many light element primary metals alloyed with a heavier element well beyond the solubility limit of the primary phase, such conditions could be met. Fig. 1 shows a columnar dendrite taken from a radiogram acquired during isothermal holding in an Al–15 wt.% Cu alloy. X-ray transmission contrast variations are clearly visible along the primary and secondary dendrite arms, reflecting dendrite thickness variations in the form of side-branching along the projected direction. Thus, if it is feasible to extract reasonably reliable estimates for the constitution of the liquid volumes sandwiched between the dendrite and the sample container, it should be possible to extract dendrite thickness information from X-ray absorption analysis. In the study reported here, efforts have been made to retrieve some of the lost spatial information from a time-resolved X-radiography sequence acquired in situ during directional solidification of an Al–Cu–Si alloy.

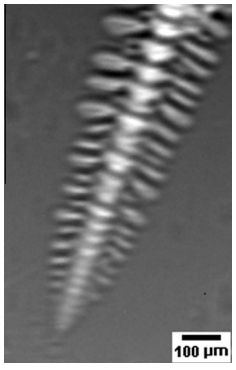

Fig. 1. Radiogram of a columnar dendrite acquired during isothermal holding in an Al–15 wt.% Cu alloy.

2. Experimental

The experiment was carried out at the hard X-ray microscope (HXRM) setup [10] at the ID6 beam line at the European Synchrotron Radiation Facility, using 14.5 keV incident monochromatic X-rays. Images over a 0.9×0.7 mm^2 field of view were captured at a 10 Hz frame rate with a 1376×1040 pixel area Sensicam-CCD camera equipped with a LAG:Eu scintillator. Directional solidification parallel ($\|$) with gravity of a 140 µm thick, 12 mm × 25 mm Al–15 wt.% Cu–9 wt.% Si–0.015 wt.% Sr sample was carried out with a Bridgman furnace used previously in a number of in situ X-radiography experiments [2]. The imposed temperature gradient was ~ 40 K mm^{-1}, while the sample pulling velocity $v_{sp} \sim 10$ µm s^{-1}.

3. Image processing and analysis

A fixed right-handed Cartesian coordinate frame is employed in all subsequent image analysis, where $\hat{e}_x$ is parallel to the incident X-ray beam, whereas $\hat{e}_y \times \hat{e}_z$ (right → left, bottom → top) spans the (y, z) projected image coordinates. For some of the calculations a microstructure reference coordinate frame $(y_d(t), z_d(t))$ is employed, which tracks a particular pixel position following the evolving microstructure, $y_d(t + \Delta t) = y_d(t) + v_y(t)\Delta t$; $z_d(t + \Delta t) = z_d(t) + (v_z(t) - v_{sp})\Delta t$ with Δt as the time step between frames, whereas v_y and v_z are the local and instantaneous solid–liquid (sl) interface velocities.

Prior to analysis, the raw radiography images were subjected to rudimentary operations, such as dark-current corrections, removal of bad pixels, noise filtering, cropping and flat fielding. These operations aim to remove or minimise raw image contrast caused by factors outside the actual metal sample arriving at a set of contrast filtered images, $I(y, z)$, as shown in Fig. 2a. The first step towards quantitative analysis is image segmentation by adaptive thresholding of the filtered images to form binary masks $M(y, z)$, discriminating 100% liquid pixels ($m_l = 1$), from pixels containing an unknown mixture of solid and liquid ($m_{sl} = 0$) (see Fig. 2b). The masks are employed in further processing to toggle between the two pixel categories in the filtered radiograms, where the operation $I_l = M * I$ leaves a liquid image with all solid-containing pixels off (with enhanced dynamic range), as shown in Fig. 2c, while $I_{sl} = (E - M) * I$, with E as a unity matrix (all pixels $e = 1$), leaves an image containing sl pixels only.

The next step is to convert the liquid X-ray transmission image into constitutional data. The transmission can be expressed via a Beer–Lambert type relationship: