A zone melting device for the in-situ observation of directional solidification using high-energy synchrotron X-rays

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Abstract

1 Directional solidification (DS) is an established manufacturing process to produce high-
2 performance components from metallic materials with optimized properties. Materials for
3 demanding high-temperature applications, for instance in energy generation and aircraft engine
4 technology, can only be successfully produced using methods such as directional solidification.
5 It has been applied on an industrial scale for a considerable amount of time, but advancing this
6 method beyond the current applications is still challenging and almost exclusively limited to
7 post-process characterization of the developed microstructures. For a knowledge-based
8 advancement and a contribution to material innovation, in-situ studies of the DS process are
9 crucial using realistic sample sizes to ensure scalability of the results to industrial sizes.
10 Therefore, a specially designed Flexible Directional Solidification (FlexiDS) device was
11 developed for use at the P07 High Energy Materials Science (HEMS) beamline at PETRA III
12 (Deutsches Elektronen-Synchrotron (DESY); Hamburg, Germany). In general, the process
13 conditions of the crucible-free, inductively heated FlexiDS device can be varied from
14 6 to 12000 mm/h (vertical withdrawal rate) and from 0 to 35 rpm (axial sample rotation).
15 Moreover, different atmospheres like Ar, N₂, vacuum can be used during operation. The device
16 is designed for maximum operation temperatures of 2200 °C. This unique device allows in-situ
17 examination of the directional solidification process and subsequent solid-state reactions by X-
18 ray diffraction in the transmission mode. Within this project, different structural intermetallic
19 alloys with liquidus temperatures up to 2000 °C were studied in terms of liquid-solid regions,
20 transformations and decompositions, with varying process conditions.

1 Introduction

Directional solidification has been applied on an industrial scale, mostly to Nickel-based superalloys, since the 1970s [1]. Especially for high temperature materials, used under creep conditions, directional solidification is a promising process. During processing, the established liquid region of the sample moves out of the heated zone and is subjected to temperature gradient. Due to that single-crystalline components or components at least exhibiting an elongated microstructure perpendicular to the temperature gradient form. These elongated microstructures can be oriented with respect to loading direction during application of the part since transversely oriented grain boundaries are a source of creep pores. By the alignment of the microstructure creep rupture can, therefore, be reduced or even eliminated [1]. For example, incorporating an aligned second phase (e.g. Cr or Mo) into NiAl [2,3] improves the creep resistance along the growth direction and increases the damage tolerance by toughening. These alloys were often produced by a Bridgman-type directional solidification apparatus, where the heated crucible rest on a water-cooled copper base. Lowering the base with a specific rate provide a movement of the melt from the hot zone into a liquid metal bath [4-7]. Another technique used is an optical floating zone furnace. Here, the heating source is a xenon arc-lamp emitting infrared radiation which is focused on the specimen enclosed in a quartz tube [2,3,8]. Ref. [9,10] produced directionally solidified specimens in a crucible-free mode by electromagnetically levitated zone process. A crucible-free process offers the advantage of no contamination/reaction from the crucible with the sample even at very high temperatures. Even though directional solidification has been applied on an industrial scale, advancing this method beyond the current applications is still challenging. Moreover, the conventional casting and directional solidification processes are not suitable for all high-temperature materials beyond Nickel-based superalloys. A limiting factor is a possible reaction of the material with the crucible at such elevated temperatures. Novel materials or changes in the components require significant changes in the process parameters, which is mostly done on a trial-and-error basis. A knowledge-based advancement of the process is difficult, as – at least to some extent – there is no possibility to characterize the production process as well as the materials behavior “in-situ”. Investigations are almost exclusively limited to a post-process characterization of the microstructures, which are often not the same as directly formed by cooling short below the solidus line. X-ray diffraction methods are suitable for in-situ studies of the DS process. With these techniques, the investigation of the liquid zone and solidification front is possible in transmission mode. Due to the high temperatures and the difficulties in handling sufficient

54 amounts of hot melt, studies of the melting and solidification behavior so far have almost
55 exclusively been performed for metals or alloys with rather low melting points, e.g. magnesium
56 or gallium-indium alloys [11-13]. Our crucible-free, inductively heated FlexiDS device allows
57 in-situ studies of the synthesis, transformation, and decomposition of various structural
58 intermetallic materials with maximum liquidus temperatures of 2200 °C via diffraction of high-
59 energy synchrotron radiation. The maximum sample diameter is 14 mm, which is a compromise
60 between possible X-ray transmission and a process that is close enough to relevant industrial
61 processes concerning temperature distribution in the solid and convection in the melt. The
62 device has been designed for the use at the P07 High Energy Materials Science (HEMS)
63 beamline at PETRA III (Deutsches Elektronen-Synchrotron (DESY); Hamburg, Germany),
64 where the combination of a high photon flux ($>10^{12}$ 1/s) and fast readout 2D detectors (15 Hz)
65 enables the study of this complex liquid-solid interactions during solidification and subsequent
66 solid-state reactions. The available detectors cover a range up to 250 Hz, corresponding to a
67 temporal resolution of 4 ms. However, 250 Hz is never needed with thick samples. The high
68 beam energy of typically 100 keV allows transmission of samples with various diameters (14
69 to 8 mm) and densities typically between TiAl and Mo-based alloys. A large portion of
70 reciprocal space can be investigated at rapid acquisition times (typically 1-30 s, depending on
71 the sample size and alloy system). This technical environment enables to investigate the local
72 evolution of the microstructure during directional growth depending on different process
73 conditions (withdrawal rate and rotation speed, rod diameter).
74 In the following, the directional solidification device and its use for characterizing the processes
75 occurring during solidification is described in detail. This provides the base for an
76 understanding of solidification and the development of alloys, which are produced by
77 directional solidification.

2 Technical layout of the FlexiDS device

78 The DS device is in essence independent and can, thus, be used either in the laboratory or it can
79 be integrated at a beamline (here: P07 High Energy Materials Science (HEMS) beamline at
80 PETRA III). In order to perform in-situ investigations during directional solidification with the
81 required flexibility, the device has to fulfill both, the requirements of the directional
82 solidification process as well as the additional demands for combining this process technology
83 with X-ray diffraction geometries.
84 A schematic drawing of the main modules of the device is visualized in Figure 1. The
85 mechanical set-up manufactured by SYSTEC Fertigungstechnik GmbH & Co. KG [14]

86 includes the following modules: I) directional solidification device on a positioning stage, II)
87 energy supply and vacuum pump, III) water battery (Figure 1). All components are designed
88 for flexible set-up and transport. Therefore, a precise positioning of the chamber with respect
89 to the X-ray beam is possible. For the directional solidification process, a clean and protective
90 atmosphere is essential to protect the material from oxidation and contamination. For the sake
91 of clarity, Figure 2 gives an overview of the complex device with all components and features.
92 Important features and parameters are compiled in the following list:

- 93 • Atmosphere: Ar, N₂, vacuum
- 94 • Heating: inductive
- 95 • Power: 12 kW (150...375 kHz)
- 96 • T_{max}: 2200 °C (tested 2000 °C)
- 97 • Rod-shaped samples from 8 to 14 mm in diameter
- 98 • Available solidification parameter:
- 99 • Vertical withdrawal rate: 6 - 12000 mm/h (6 mm/h increments, tested so far 6 -
100 360 mm/h)
- 101 • Axial sample rotation 0 - 35 rpm (1 rpm steps), synchronous rotation or counter rotation
- 102 • Temperature observation: two pyrometers positioned at the liquid zone (in the coil) and
103 10 mm above

104 The manually controlled power of 12 kW is spread from 0 to 100 % and can be adjusted in
105 0.1 % steps, which enables the user to reach and maintain the sample in the liquid state.

Figure 1: Schematic of the modules of the setup: I) Directional solidification device on positioning stage, II) Energy supply and vacuum pump, III) Water battery. The person is displayed here to estimate the size and usability of the device.

Figure 2: Overview of the zone melting device with its components.

2.1 Process chamber

106 A visual inspection of the process chamber interior is possible via multiple windows and a lamp
107 illuminating the chamber. Furthermore, the process chamber can be monitored via two high-
108 resolution cameras suitable for high temperature image acquisition (detailed description in
109 section 3). These cameras may also be used to observe the liquid zone of the sample. Two
110 pyrometers detect the temperature of the rod inside the coil and at an adjustable distance above
111 the coil (typically 10 mm above the induction coil). With this arrangement, the temperature can
112 be measured in the liquid zone and in 10 mm distance, usually already in the solid state of the
113 sample. With the assumption of linear temperature decrease, a temperature gradient can be
114 determined. The temperature evolution plays an important role in terms of process control and
115 power adjustment. Spare flanges can be used for additional measuring devices or other
116 attachments. One is located at the chamber door (DN 25 ISO-KF) (Figure 2) and a second at
117 the backside of the chamber (DN 40 ISO-KF) (Figure 3). Figure 3 shows the interior view of
118 the chamber. For the sake of safety and sealing, the process chamber door is equipped with two
119 locks. Moreover, a door protection switch is attached which prevents that process gas flow and
120 heating is activated if the process chamber door is open.

Figure 3: Interior of the process chamber: The water-cooled chucks and the high frequency (HF) coil as the heating source are highlighted. The X-ray beam enters the chamber on the right side, interacts with the sample and the diffracted beam leaves the chamber on the left side through the larger window.

2.2 Sample holder and induction coil

121 The sample passes through the induction coil (Figure 3) and is fixed in the upper and lower
 122 drive shaft by clamping jaws. 10 mm of the sample are needed on both ends for mounting the
 123 sample in the clamping system. Together with a typical safety distance of 20 mm from the coil
 124 below and above preventing induction in the clamps, the sample length is calculated. The upper
 125 chuck is tightened manually while the lower one has a pneumatic system in order to allow the
 126 sample to expand during heating up and to tighten it right before the liquid zone forms. With
 127 the two independent drive shafts, a vertical movement of the sample and synchronous or counter
 128 rotation can be applied. The induction coil displayed schematically in Figure 4a was specially
 129 designed for in-situ experiments and is suitable for the above-mentioned sample geometries.
 130 An integrated water circuit achieves cooling of the coil, chucks and housing. The conical recess
 131 in the coil prevents cutting off for the diffracted beam to pass (Figure 4a). Figure 4b gives an
 132 insight into the chamber during heating. For the in-situ experiments, the samples were usually
 133 moved upward (lifted) through the coil due to the position of the beam and the recess of the
 134 coil. Thus, the upper sample part represents the directionally solidified part and the lower part
 135 is called the feed, which is in the as-cast condition. For the results shown in section 5, both parts
 136 of the rod are rotated with the same speed and direction, but these two parameters can be also
 137 varied independently.

Figure 4: a) Detail of the specifically designed HF coil, b) HF coil and a heated sample during processing. Arrows indicate the process movement in terms of withdrawal rate and rotation speed.

2.3 Positioning stage

138 In order to move the FlexiDS process chamber with respect to the incident beam parallel as
139 well as transverse to the samples' growth direction, the system is mounted on a positioning
140 stage (Figure 5). Once placed with support rollers at the final position, the device is set on the
141 support stands, which are located at the corners of the stage. A spirit level facilitates the
142 adjustment and ensures the horizontal alignment. The movement range is ± 50 mm in growth
143 direction and transverse to growth direction. The smallest positioning increment is 0.05 mm
144 allowing an accurate positioning with respect to the incoming beam.

Figure 5: Positioning stage, range of motion is ± 50 mm parallel and transverse to growth direction.

3 Workstations and control software

145 Two workstations, a mobile panel and a PC workstation, are connected to the device. The
146 control software is implemented by iTG Induction systems [15]. Generally, two modes of
147 operation are possible: “manual mode”, mainly operating the mobile panel, and “auto mode”,
148 mainly suited for the PC control during in-situ experiments.

149 The mobile panel next to the chamber allows fast and precise setting up and positioning of the
150 sample. In order to achieve the correct position of the sample with respect to the induction coil,
151 the upper and the lower driving shaft are controlled individually. Apart from the actuators, the

152 coil power, the vacuum pumps and the gas flow can be controlled from the mobile panel in
153 “manual mode”.

154 The spatial separation of device and PC process control is necessary for the in-situ observations
155 during directional solidification. By activating the “auto mode” the vacuum pumps
156 automatically turns on and the chamber is evacuated to $2 \cdot 10^{-5}$ mbar, once the sample is
157 placed in the device and the door is properly closed. In a second step, the process chamber is
158 purged with Ar. During the DS process, Ar flow is active. The PC workstation controlling the
159 device consists of process parameter control like withdrawal rate, rotation speed, coil power as
160 well as movement of the positioning stage. A detailed camera view of the sample (Figure 6)
161 and pyrometers are incorporated in the PC controlling unit. Within the “auto mode”, the process
162 control software saves all parameters and the corresponding changes with timestamps in a log
163 file.

164 For in-situ observations during the process, a second person is recommended to adjust the beam
165 and to run the recording of the diffraction data.

166 The reaction of the sample to power adjustments can be observed with the high dynamic range
167 cameras (hema electronic GmbH, Germany, type **seelector**ICAM weld [16]). One is placed to
168 give information from below the coil and the second one to show a view from above the coil.

169 The cameras are sensitive to temperatures below 1000 °C and the contrast can be adjusted for
170 an optimal visual representation. The lenses C2514-M (KP)/Ricoh FL-CC2514-2M - 1.4/25mm
171 from Pentax ensure a balance between clarity and details of the liquid zone. The pyrometers
172 utilized for our device measure temperatures between 1000 and 3000 °C (LumaSense
173 Technologies GmbH, Germany, type IMPAC pyrometers ISQ 5 [17]). In the “LumaSense”
174 software data of both pyrometers is plotted in a temperature vs. time diagram. Temperature
175 measurement is necessary to control the process and gives information about the temperature
176 gradient within the sample. Examples of the detailed camera view during the process are given
177 in Figure 6a and b, from below and from above the coil, respectively. Both information are
178 crucial for process control. In order to establish a stable liquid zone sufficient power and
179 overheating is required. When the power level is too high, depending on the liquid surface
180 tension, locally an expansion of the liquid zone arises (Figure 6a). The surface features in Figure
181 6b are oxide scales forming due to the oxygen in the sample. It appears brighter than the liquid
182 beneath. Thus, these oxides visualize the movement and the overheating of the liquid zone and
183 may serve as indicators during the process.

Figure 6: Visualization of the sample from two different points of view: (a) below the coil and (b) above the coil. The insert is a so-called region of interest (ROI). The settings only for this region is can be adjusted variable separately from the general settings of the image.

4 System integration at the P07 High Energy Materials Science Beamline

184 The modules are placed in the experimental hutch 3 (EH3) at the beamline P07 at DESY in
 185 Hamburg as illustrated in Figure 7a. The X-ray beam transmits the chamber from the right to
 186 the left in Figure 7a and b. After the interaction with the sample, the diffracted beam leaves the
 187 chamber through the larger window on the left side of the chamber and is captured by the
 188 detector placed in a certain distance from the sample. It is noteworthy that because of the
 189 crucible-free set-up and the recess in the coil the incident beam is only diffracted by the sample
 190 (without any alien reflections). This facilitates the evaluation of the diffraction data. Diffraction
 191 resulting from the windows are negligible. The on-site infrastructure, which allows beam and
 192 detector control, is used for the X-ray diffraction data acquisition.

193 The X-ray energy at P07 is tunable from 30 keV to 200 keV with its main optics consisting of
 194 a water-cooled double crystal monochromator in horizontal Laue scattering geometry. The
 195 energy resolution ($\Delta E/E$) ranges from 1×10^{-3} down to 7×10^{-5} by changing the curvature of
 196 the bent Si (111) crystals in Rowland geometry and/or inserting a channel-cut crystal. The spot
 197 size can be varied from $1 \text{ mm} \times 1 \text{ mm}$ (unfocused beam) down to $2 \mu\text{m}$ vertical $\times$ $30 \mu\text{m}$
 198 horizontal (focused beam via Al Compound Refractive Lenses) in high-beta mode or up to
 199 0.9 mm vertical $\times$ 6 mm horizontal in low-beta mode, where the beta mode is switched by
 200 changing the beta function at the undulator source position. The maximum flux currently
 201 reaches 7×10^{11} ph/s at 100 keV [18,19].

Within the project, five material systems, as indicated in the first column of Table 1 and several alloy compositions of each system were investigated. The different content of heavy elements in the systems provoke significantly different transmission behavior. Thus, exposure times have to be adapted to the respective material (Table 1). The signal to noise ratio obtained by maximum intensity and background for all investigated alloy systems is given in the last column of Table 1. A beam size of $1\text{ mm} \times 0.5\text{ mm}$ was used to achieve sufficient grain statistics and conveniently short exposure times. A Perkin Elmer XRD 1621 solid-state detector with

209 2048×2048 pixels and a pixel size of $200 \mu\text{m} \times 200 \mu\text{m}$ was used for all measurements. As
210 seen in Table 1 a photon energy between 98 and 103.6 keV was used, corresponding to
211 wavelengths between 0.126 and 0.119 Å. The detector was located in approximately 1.9 m
212 distance from the sample. The resulting maximum 2θ angle was 6° and the step size is 0.006° .
213 Si and CeO_2 powder, were used as reference to calibrate the measurements.

Table 1: Experimental parameters for the investigated materials.

Material	Photon energy [keV]	Beam size [b mm $\times$ h mm]	Exposure time [s]	Signal to noise ratio [a.u.]
Fe-Al [20]	98	1 x 0.5	2 – 3	60 – 120
Nb-Si-Cr [21]	98	1 x 0.5	20 – 30	28 – 34
NiAl-Cr,Mo[22]	98 – 103.6	1 x 0.5	1 – 12	75 – 225
Mo-Si-B [23]	98 – 103.6	1 x 0.5	6 – 32	22 – 172
TiAl [24]	98 – 103.6	1 x 0.5	2 – 6	105 – 135

5 Examples of in-situ X-ray diffraction experiments during directional solidification

214 In the following, two examples for in-situ investigation of directional solidification of high
215 melting point two-phase intermetallic materials are given. The first case focuses on the Ti-Al
216 system, or more precisely on so-called γ -TiAl-alloys which have been commercialized already
217 in investment cast, isothermally forged as well as in additively manufactured (by EBM) form
218 [25-28]. DS might enable a unique combination of high creep strength and ductility [29], but
219 the decisive processing parameters are still unknown. The second case considers a high Al-
220 containing two-phase FeAl alloy, which undergoes a high temperature eutectoid decomposition
221 reaction upon solidification via a peritectic reaction. Here, the overarching scientific question
222 is whether the eutectoid lamellae can be properly aligned during DS in order to also create
223 creep-resistant microstructures at elevated temperatures. All chemical compositions mentioned
224 in what follows are given in at.%.

5.1 The Ti-Al system

225 As mentioned above DS is an attractive processing method for γ -TiAl-alloys because it allows
226 to align the constituting phases of the material [29]. However, the knowledge about suitable
227 processing conditions is limited. For example, the effects of different alloy compositions on the
228 solidification process are still not well understood. In-situ investigations are a powerful tool to
229 get a deep insight into the solidification of TiAl alloys. All the more as it is difficult to yield
230 information on the solidification process from investigations at room temperature because

subsequent solid-state phase transformations will mask the high-temperature situation to a significant extent.

An example of investigations on TiAl alloys is an experimental proof of the predictions for solidification [24], which were made based on the „nucleation and constitutional undercooling“-model of Hunziker et al. [30]. If correct, a strong dependence of the type of the solidifying phase as well as its morphology on the G/v ratio should be found (G : temperature gradient, v : withdrawal rate). Solidification experiments with varying withdrawal rate between some mm/h up to some hundreds mm/h were performed with different binary TiAl-alloys. Ti-45Al solidifies via the β -phase (see phase diagram Figure 8). For this alloy a change from planar to dendritic solidification with increasing withdrawal rate (decreasing G/v ratio) is predicted (Figure 9a). The other alloy, Ti-48Al, also changes from planar to dendritic solidification mode with increasing withdrawal rate, but the solidifying phase is additionally predicted to change from α -phase to a two-phase α/β mixture. From the diffraction ring patterns as shown in Figure 9b and c, it is obvious that for Ti-48Al the change of the solidifying phase from α -phase to a two-phase α/β mixture with increasing withdrawal rate is experimentally verified. For a withdrawal rate of 36 mm/h only reflections of melt (L) and α -phase were present. α - and β -phase solidify simultaneously from the melt for an increased withdrawal rate of 360 mm/h, however the change in phase constitution occurs at a significantly lower G/v value as obtained by the model. The numerical difference might originate from improper model parameters, but this question is beyond the scope of the present work. Also other predicted effects such as a change of the solidification morphology from planar to dendritic with increasing withdrawal rate were confirmed in Ti-48Al as well as in Ti-45Al, although in the former case it occurred again at a lower G/v value than obtained by the model (Figure 9a). In summary, by in-situ studies it can easily be analysed at which conditions certain phases and morphologies will form in TiAl alloys during solidification without being constrained by indirect conclusions as it is the case in post process investigations.

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Figure 8: Section of the equilibrium Ti-Al phase diagram [31]. Reprinted from Acta Metallurgica, Vol. 37, C. McCullough, J. J. Valencia, C. G. Levi, R. Mehrabian, Phase equilibria and solidification in Ti-Al alloys, pages 1321-1336, Copyright (1989), with permission from Elsevier.

Figure 9: a) Predicted solidification morphology and solidifying phases in dependence on G/v and alloy composition. Resulting G/v values in the experiments are indicated by dots. Diffraction pattern of the solidification front of Ti-48Al at different withdrawal rates, b) 36 mm/h and c) 360 mm/h.

5.2 The Fe-Al system

257 In Fe-61Al, a eutectoid decomposition Fe_5Al_8 (Cu_5Zn_8 structure type) into FeAl (CsCl structure
258 type) and FeAl_2 (triclinic, no other known structure of this type) occurs. The high temperature
259 phase Fe_5Al_8 results from a peritectic reaction and, therefore, a two-phase field must be passed
260 in order to obtain this phase during the solidification of this specific alloy. The according section
261 of the equilibrium phase diagram is displayed in Figure 10a. The solid-state decomposition
262 results in a lamellar microstructure being potentially promising with respect to its creep

263 resistance in conjunction with the generally low density and very good oxidation resistance of
264 high Al containing iron aluminides.

265 Diffraction patterns obtained during directional solidification of this alloy within the FlexiDS
266 device are depicted in Figure 10b to e. The diffraction pattern in Figure 10b was obtained at the
267 solidification front, Figure 10 c,d and e in 3.5, 4.5 and 5.5 mm distance from the solidification
268 front respectively. The corresponding temperatures are indicated in Figure 10a. As seen in
269 Figure 10c, only Fe_5Al_8 appears within the single-phase region. Retardation of the phase
270 formation and excess FeAl_2 due to the slow peritectic reaction were not observed. The 2d
271 diffraction pattern in Figure 10 c, d and e were converted to 1d. The corresponding 1d
272 diffraction pattern are presented in Figure 11. Lattice parameters were found to be similar to
273 those reported by Stein et al. [33] for cast material. In contrast to expectations in Ref. [20] there
274 is no evidence for preferential orientation of the high temperature phase with respect to the
275 solidification direction even though a rather large range of withdrawal rates was tested, namely
276 6 to 240 mm/h. Nevertheless, columnar Fe_5Al_8 grains form at high withdrawal rates which
277 results in columnar eutectoid colonies after completion of the eutectoid reaction. For
278 withdrawal rates higher than 120 mm/h, inhomogeneous intensity distributions for the (001),
279 (010), and (011) diffraction rings of FeAl_2 indicative of a crystallographic texture are observed
280 in Figure 12a. Indeed, a majority of the lamellae in the eutectoid microstructure are aligned
281 with respect to the temperature gradient as seen in Figure 12b. For a single orientation of Fe_5Al_8 ,
282 twelve different orientation variants of the lamellae could be expected based on the distinct
283 orientation relationship of FeAl and FeAl_2 [20]. Obviously, high withdrawal rates lead to a
284 variant selection of the colonies in the temperature gradient during the eutectoid reaction even
285 though the parent phase does not exhibit preferential orientation. Without the application of in-

286 situ observation of the solidification and cooling process, this information would not have been
287 accessible.

Figure 10: (a) Section of the equilibrium Fe-Al phase diagram close to the eutectoid decomposition of Fe₅Al₈ into FeAl and FeAl₂ [32], (b) to (e) diffraction patterns obtained at four different temperatures as highlighted in (a). (a) is taken

from X. Li, A. Scherf, M. Heilmaier, F. Stein, The Al-rich part of the Fe-Al phase diagram, J. Phase Equilibria Diffus. 37 (2016) 162-173; licensed under a Creative Commons Attribution (CC BY) license.

Figure 11: Diffraction patterns from Figure 10c to e converted to 1d diffraction patterns.

Figure 12: (a) Diffraction pattern of Fe-61Al at room temperature subsequent to directional solidification at 120 mm/h. Slight crystallographic texture is obtained for FeAl₂ indicated by inhomogeneous intensity distribution along the (001), (010), and (011) diffraction rings highlighted with arrows. (b) Scanning electron micrograph (backscatter electron imaging) of a longitudinal section of the directionally solidified material (bright phase is FeAl and dark phase is FeAl₂).

6 Summary

288 We introduced the new state-of-the-art FlexiDS device for time-resolved in-situ investigation
289 of directional solidification processes using high-energy synchrotron radiation. The system is a
290 valuable addition to the infrastructure of PETRA III at DESY, being specifically designed for
291 use at P07 HEMS beamline. The apparatus is capable of investigating liquid-solid regions,
292 transformations, and decompositions in various structural intermetallic materials. Moreover,
293 we could show the wide operation range by means of sample geometry, densities and alloy
294 compositions as well as process parameter like withdrawal rate. Even during experiments which
295 exceed a liquidus temperature of 2000 °C, the device operated stable and satisfactory diffraction
296 pattern were obtained. Trial tests of time-resolved processes of TiAl and FeAl alloys were
297 presented in this work. Further detailed experimental results are forthcoming and will be
298 published elsewhere, for instance Ref. [22]. Not being installed at the beamline, the FlexiDS
299 device is available at the Helmholtz-Zentrum Geesthacht. Possible trial tests on new alloys can
300 be performed there without using high-energy synchrotron radiation in order to get used to the
301 device. We strongly welcome and encourage collaboration from the materials science research
302 community in order to maximize the potential of this unique in-situ directional solidification
303 device for improving understanding of fundamental solidification processes.

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Data availability statement

310 Data available on request from the authors. Please contact alexander.kauffmann@kit.edu.

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- 1 process chamber
- 2 high resolution camera
- 3 pyrometer
- 4 gas distribution Nitrogen
- 5 oil reservoir
- 6 drive unit upper punch
- 7 gas distribution Argon
- 8 pyrometer
- 9 water supply
- 10 drive unit lower punch
- 11 power and network cables
- 12 podium
- 13 positioning table
- 14 vacuum hoses
- 15 terminal box

Upper chuck

Spare flange

Incident beam

Heating
source:
HF coil

Diffracted beam

Sample

Exit window

Lower chuck

25 mm

Intensity / a.u.