

## **In Situ Microscopy on the Melting and Cooling Behavior of an Al-Si 12 Alloy Using ZEISS ZEN 2 core**



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Authors: Jörg Maier, Tim Schubert, Dr. Lisa Weissmayer,  
Dr. Timo Bernthaler, Prof. Dr. Gerhard Schneider  
*Aalen University, Materials Research Institute*

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## Introduction

The steadily increasing quality requirements mandatory in the foundry industry, which also requires a high degree of economic efficiency, necessitate specific requirements in terms of material characteristics and operational behavior. This applies in particular to lightweight alloys as well as piston alloys made from aluminum-based castings. Some of the most important aluminum casting alloys are the hypoeutectic aluminum-silicon alloys, which have a silicon content of 5–12 weight-%. These casting alloys are characterized by an excellent mold filling and solidification behavior. These net-shaped casting components are structurally feasible and can be used to meet various technical requirements thanks to the properties of the cast aluminum alloys. The realistic stress profile requires the creation of a homogeneous, fine-grained, and defect-free structure with low density and strength – even at high melting temperatures. In situ observation of phase transitions and solidification processes at defined temperatures is therefore an excellent method of observing casting and heat treatment processing of alloys. It also provides a better understanding of structural formation, making it possible to make predictions about technical casting processes involving various composite alloys.

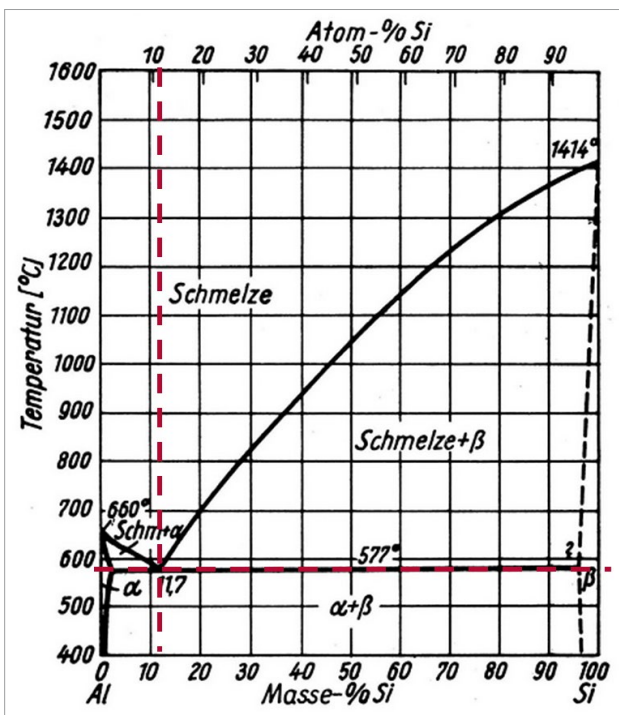
For example, observe the spatially and temporally resolved melting and cooling behavior of a eutectic Al-Si 12 alloy. The experimental hardware setup for the experiments included:

- ZEISS Axio Imager.Z2m light microscope
- ZEISS 20× long-distance Epiplan lens
- Linkam TS1500 heating stage with vacuum pump and inert gas (optional)
- Linkam T95 controller control panel

The Linkam TS1500 heating stage is suitable for studying a variety of materials, including ceramics, metals, and polymers, under temperature. Samples with a limiting magnitude of a maximum of 7 mm in diameter and 6 mm in height can be heated to a maximum 1.500°C in the ceramic sample cup. The T95 controller is used to reach maximum heating and cooling rates of approximately 200 K/min. Using a rotary vane pump, the heating stage can be operated in a vacuum state at  $10^{-3}$  mbar. Activation can be controlled manually via the T-95 controller or using the new ZEISS ZEN 2 core software. At this point, the user has two options for conducting the experiment. In experiment mode the user can set up a temperature curve for the Linkam stage which comprises individually defined temperature ramps connected to each other. The rate of heating and cooling, the dwell time or final temperature can be set for every temperature ramp individually. For each temperature ramp, the user can define a condition for when an image should be acquired with the microscope. This can either be each time a defined time interval has elapsed or each time the temperature has changed by a defined value. Individual blocks can be predefined using the above parameters that occur sequentially while the experiment is being conducted. In manual mode the user can manually control the Linkam Heating Stage and the ZEISS Axio Imager.Z2m light microscope by using ZEISS ZEN 2 core. This mode allows the user more flexibility in the experiment setup to customize it by making changes while it is being performed. As the sample changes elasticity with increasing heating or cooling, the lens focus requires monitoring and adjustment during the experiment.

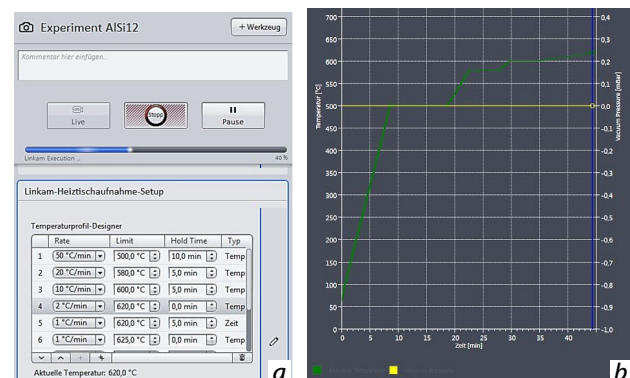
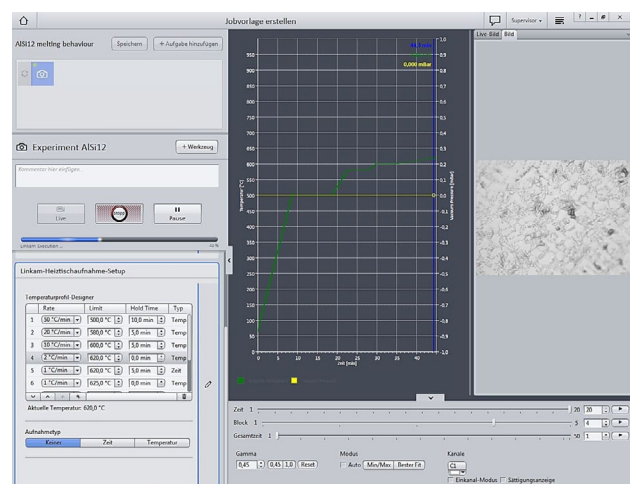
### Experimental Procedure and Results

The aim of examining the Al-Si 12 alloy was to observe its transformation behavior at the eutectic point of 580°C (Figure 1 → intersection of the red dashed lines) while heating and subsequent cooling. In this case, the entire system was brought to a vacuum state of around  $2 \times 10^{-3}$  mbar by purging it with inert gas. The experiment was performed using the “experiment mode” in ZEISS ZEN 2 core; thus the different heating rates, holding points and dwell times – including the time intervals between image acquisitions – were determined by the “heating stage control” control setup (Figure 2a). The heat ramp diagram (Figure 2b) illustrates the heating rates, breakpoints and dwell times during the experiment. First, the sample was heated up quickly at a rate of 50 K/min to 500°C and then held there for three minutes so that the sample could settle into this temperature. The process was repeated once more with a heating rate of 20 K/min for the actual target temperature according to the phase diagram of 580°C.

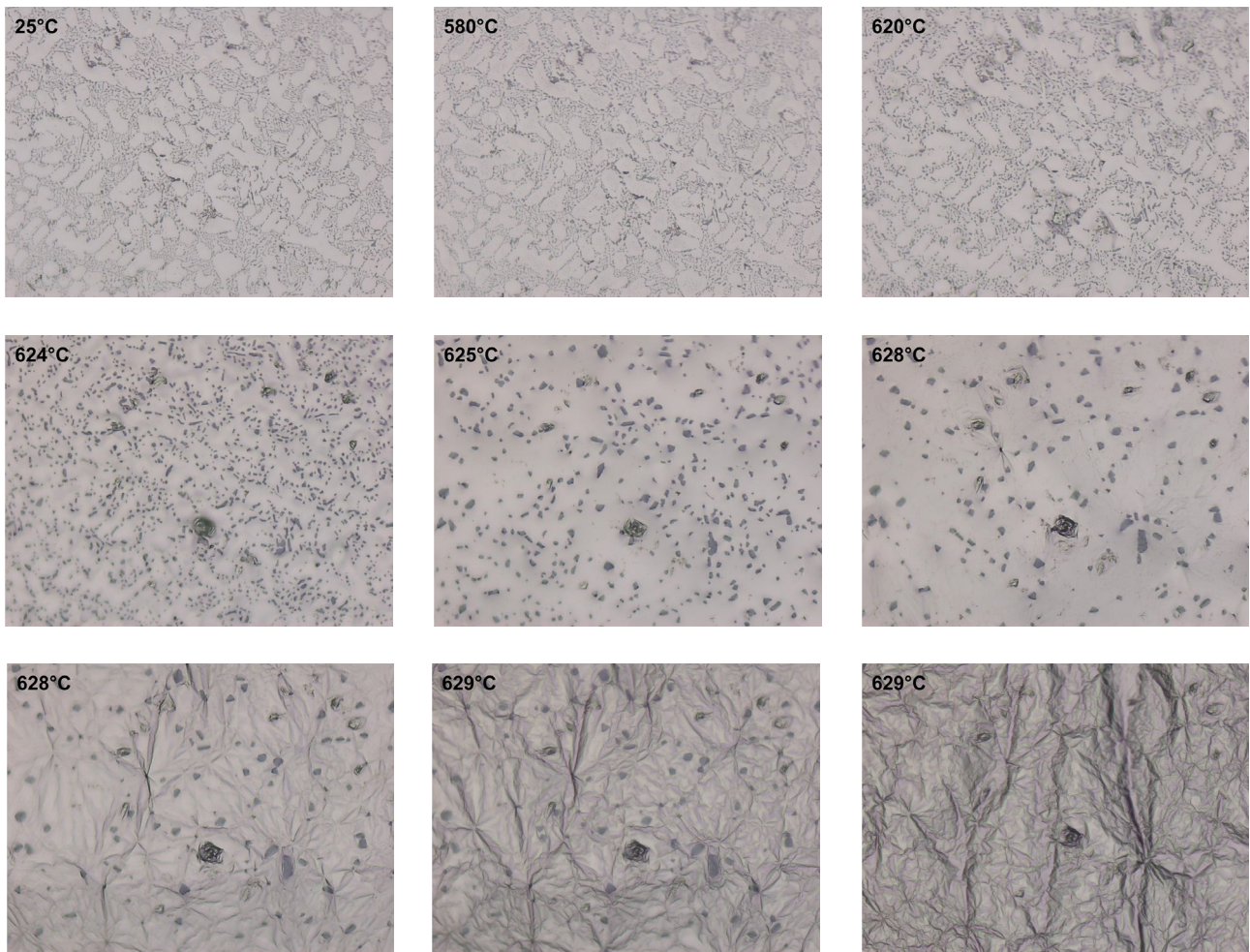


**Figure 1:** Phase Diagram (temperature in °C on the ordinate and mass-% resp. atom-% on the abscissa) for Aluminum-Silicon eutectic at 11.7 mass-% Si [1]

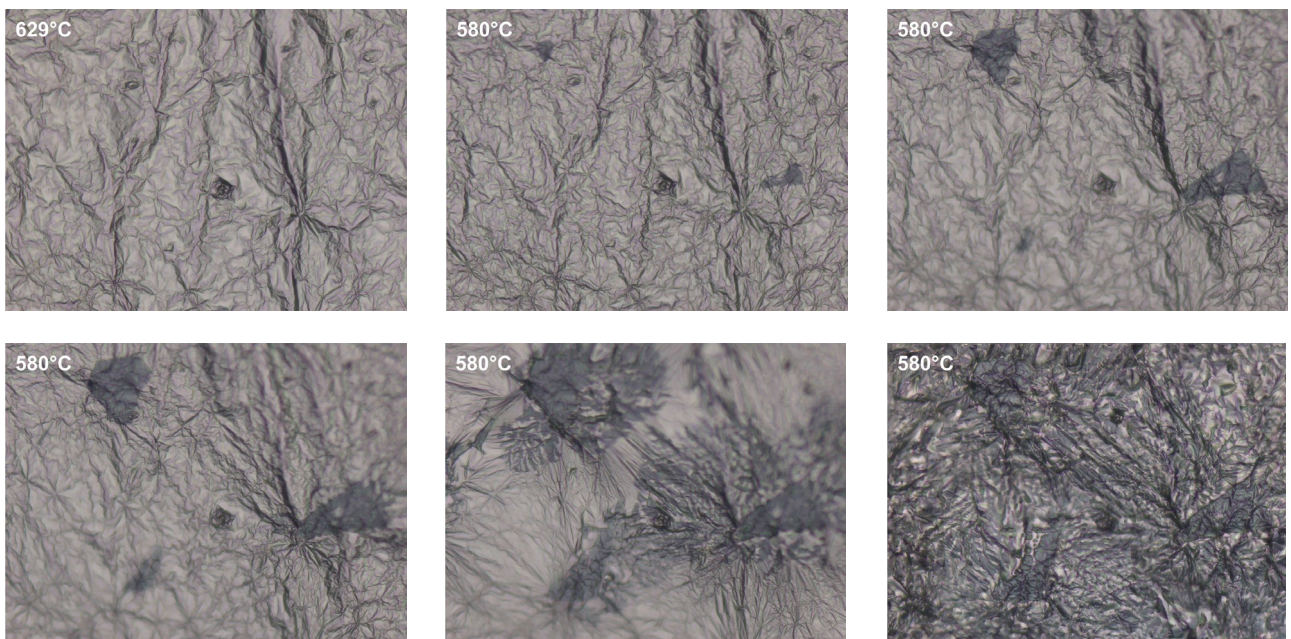
As no conversion reaction manifested itself in the form of a melting process (Table 1; Figure 2), heating up continued at a heating rate of 10 or rather 2 K/min and was then maintained at 610°C and 620°C for two minutes each. Conversion reactions were first observed at 620°C. In order to add further energy to the melting process and give the conversion more time, the sample was further heated to 629°C at 1 K/min. At this point, the aluminum (white phase) slowly melted and dissolved the silicon (dark phase), which, at the same time, coarsened due to diffusion processes and dissolved in the molten metal towards the end of the process. The temperature dependence of the melting process is microstructurally depicted in Figure 3.



**Figure 2:** “Linkam experiment mode” in ZEISS ZEN 2 core for the Al-Si 12. Alloy melting investigation. Detailed picture of “heating stage control” setup (a) and heating ramp diagram (b)



**Figure 3:** Heating Process for an Al-Si 12 Alloy to 629°C



**Figure 4:** Cool-Down Process for an Al-Si 12 Alloy Starting at 629°C

The cooling process (Figure 4) starts at 629°C with a constant cooling rate of 5 K/min down to 580°C. After six minutes of dwell time, a new conversion reaction begins as the silicon primarily and dendritically precipitates. A subsequent cool-down that is even more rapid, going from 580°C at 50 K/min, shows no further changes in the structure. All pictures taken during the experiment can be edited afterwards and it is also possible to create a video. The entire experiment with the complete setup can be saved for further investigations with the same material.

**Video for the Melting and Cooling Processes for the Al-Si 12 Alloy:**



**Reference:**

[1] Schumann, Hermann: Metallographie; 13. Auflage, Deutscher Verlag für Grundstoffindustrie GmbH, Leipzig, 1991, S. 682



**Carl Zeiss Microscopy GmbH**  
07745 Jena, Germany  
microscopy@zeiss.com  
www.zeiss.com/microscopy



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