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Differential fast scanning calorimetry as analytical tool for mimicking melting and solidification in additive manufacturing

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Abstract: This paper shows a method to deepen the knowledge about the rapid solidification of metal alloy powders via in-situ investigation and to guide the proper parameter selection for rapid solidification process methods, e.g. laser or electron beam melting in additive manufacturing (AM). This allows developing microstructure maps to establish regimes of alloy composition and undercooling required for design of materials with improved mechanical properties. Differential fast scanning calorimetry (DFSC) characterization and microstructure analysis carried out on the example of Al-Si alloys, as important engineering materials often used in AM, will improve understanding of rapid solidification processes and microstructure formation.

I. Introduction

Additive manufacturing (AM) of metals for instance by laser beam melting (LBM) holds great potential for improving materials efficiency, reducing life-cycle impacts, enabling greater engineering functionality and allowing fast reaction on customer's demands [1]. For example, the application of LBM components in biomedical engineering has the potential to provide individual implants for the unique therapy situation of single patients in short times [2].

Aluminum-based alloys, like Al-Si alloys, are important engineering materials in general and are often used for LBM. They are not used as implant materials, but the presented method can be transferred to implant materials, like Mg- or Ti-alloys. Local cooling rates during solidification in AM agree with those of other rapid solidification processes (RSP). By using rapid solidification techniques, e.g. atomization, melt spinning, spray forming and splat quenching, cooling rates from 10^3 K/s up to 10^7 K/s can be achieved [3]. Numerous ex-situ studies [3, 4] have been reported on the rapid solidification of Al-Si alloys by melt spinning, atomization, laser melting, spray forming, etc., which significantly modifies the microstructures. However, in-situ investigation of rapid solidification is missing.

Recently, the Competence Centre °CALOR at University of Rostock has developed a non-adiabatic differential fast scanning calorimeter (DFSC) with a wide range of heating, and more important, controllable fast cooling rates [5]. This new thermal analysis device provides the opportunity to investigate the fundamental issues related to rapid solidification mentioned above. Until now, to the best knowledge of the authors, this is the only analytical technique that is capable of applying both controllable heating and cooling at rates up to 10^6 K/s [5]. This interval is of particular interest for the rapid solidification process of micro-sized metal alloy as well as polymer particles because it covers the technologically relevant range of the heating and cooling rates for common AM technologies. Based on the calorimetric method developed for the study of metal particles, the solidification process of aluminum-based alloy powder-particles, i.e., AlSi10Mg, was studied, for the very first time, under AM relevant heating and cooling rate conditions.

II. Material and methods

A differential fast scanning calorimeter (DFSC) based on thin film sensors (XI-415 high temperature sensor, Xensor Integrations, The Netherlands) was used for the measurements of the thermal effects associated with the melting and solidification of AlSi10Mg powder particles. The sensor consists of an amorphous silicon-nitride membrane with a film-thermopile and a resistive film-heater placed at the center of the membrane, as shown in Fig. 1(a). To improve heat contact between sample and membrane, silicone oil was used. The sample had to be placed in the center of the heated area to avoid probing the strong temperature gradient outside this area. The sample was heated under an air atmosphere from 47 to 627 °C. Temperature calibration of the DFSC was performed prior to the experiments at different heating rates by measuring the onset melting temperatures of indium, tin, and zinc and the solid-state transition temperature of K_2SO_4.

III. Results and discussion

The typical temperature profile of linear heating/cooling experiments and a typical measurement curve are shown

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in Fig. 1(b). A series of DFSC heating and cooling experiments was conducted, applying cooling rates as high as 8×10^4 K/s. The melting and solidification peaks of one particle with the diameter of 12 µm can be observed at the heating and cooling rate of 2000 K/s, as indicated in Fig. 1(b). The differential fast scanning calorimeter traces revealed that the material undergoes a two-stage melting and solidification processes depending on heating and cooling rates. This may be due to the subsequent solidification of the Al-dendrites and the eutectic phase in this hypoeutectic alloy. In this research, the maximum temperature at high heating rates was extended to above 727 ºC, which is typically enough to melt most of the aluminum-based alloys used in AM technologies. Subsequently the samples are quenched with controllable cooling rates. In first preliminary experiments, we focus on the start points of the first melting stage and the solidification peaks, i.e., the onset melting temperature T_m and the onset solidification temperature T_s. In Fig. 2(a), the solidification temperature and undercooling ($\Delta T = T_m - T_s$) were determined at different cooling rates from 500 to 8×10^4 K/s for the particles with different sizes. As expected the undercooling increases with increasing cooling rate and decreasing particle size.

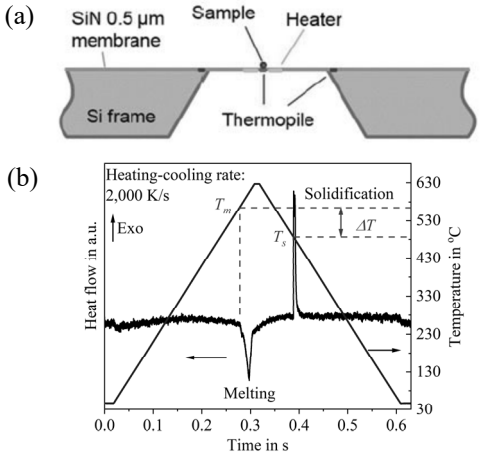

Figure 1: (a) The schematic cross-section of the sensor with sample (not to scale) [6]. (b) Typical temperature profile of linear experiments and measurement curve of one single AlSi10Mg particle. T_m – melting temperature, T_s – solidification temperature, ΔT – solidification undercooling.

Metallographic examination was performed by analyzing individual solidified particles from DFSC by optical microscopy (OM) and scanning electron microscopy (SEM). Thereby, solidification structures on the nanometer scale can be investigated. Fig. 2(b)-(c) shows eutectic like features with dimensions of a few 100 nm. The small bright spots very likely are preparation artefacts and will be removed by further improving the preparation procedure.

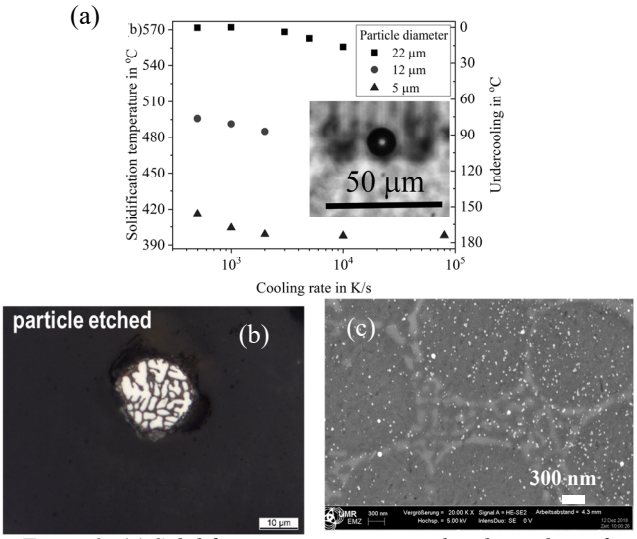

Figure 2: (a) Solidification temperature and undercooling of single AlSi10Mg particles with different sizes for a series of cooling rates. The inset shows the particle with the diameter of 12 µm on the sensor. (b)-(c) Example for OM and SEM investigation of individual metal particles: etched particle in OM and particle in SEM, respectively.

IV. Conclusions

Rapid solidification of single micro-sized aluminum-based alloy particles were successfully in-situ characterized, by application of DFSC combined with microstructure characterization. DFSC characterization carried out on AM related materials, will improve understanding of rapid solidification process under AM conditions. The presented method can be transferred to implant materials, like Mg- or Ti-alloys.

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Author's statement

Conflict of interest: Authors state no conflict of interest. Informed consent: Informed consent has been obtained from all individuals included in this study.

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