SURFACE INOCULATION OF ALUMINIUM POWDERS FOR ADDITIVE MANUFACTURING GUIDED BY DIFFERENTIAL FAST SCANNING CALORIMETRY

New materials and processes enable saving weight of parts and components and thereby saving energy. To develop and modify materials to make them suitable for additive manufacturing, the Chair of Materials Science (LWK) with the Direct Manufacturing Research Center (DMRC), the Chair of Technical and Macromolecular Chemistry (TMC) and the Competence Center °CALOR as part of Rostock University are cooperating within the Special Priority Program (SPP) 2122, promoted by the German Research Foundation (DFG). Together, they face the challenge to process hard to weld materials like high-strength aluminum alloys by using powder bed-based laser beam melting (PBF-LB/M). One approach in this project is the modification of initial state powder with nano-size particles.

PROJECT OVERVIEW

DURATION

10/2018 – 09/2021

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- Chair of Technical and Macromolecular Chemistry (TMC)
- Competence Center °CALOR, Rostock University
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Objective

The combination of lightweight materials and laser beam melting (LBM) is well suited to realize new opportunities to achieve a lightweight design. In particular, high strength aluminum alloys like EN AW-7075 (AlZn5,5MgCu) and EN AW-7021 (AlZn5,5Mg1,5) offer great potential for lightweight applications. Unfortunately, they are susceptible to pores and hot cracking during welding processes and therefore in LBM, too. Up to now, this behavior prevents their using application in additive manufacturing (AM). One solution to counteract this challenge is a modification of the aluminum powder with nanoparticles. This effect reduces or avoids the issues mentioned above and makes sure the processability of these alloys via LBM.

Approach

Based on previous research and experiences, grain refiner such as Al-5Ti-1B is used in continuous and shape casting to minimize cracking issues. In this case, Al_3Ti dissolves in the melt and TiB_2 conduce to nucleation. For application in AM, TiC and Ti_2B as nano-sized particles are applied to achieve an effective grain refinement.

With parameter studies, the appropriate adjustments of factors like laser power, scan speed, hatch distance and layer thickness are determined to achieve a high relative component density. Subsequent the micro- and nanostructure, grain orientation and corrosion behavior have been analyzed. For this, FE-SEM, TEM, XPS as well as optical spectroscopy and techniques like potentio-dynamic polarization are deployed.

The melting and cooling behavior of individual powder particles for extremely high heating and cooling rates and thus the effectivity of the surface covering grain refiner is investigated by CALOR with Differential Fast Scanning Calorimetry (DFSC). In this project, TMC has developed wet-chemical procedures to decorate the initial state powder with grain refiner. Depending on the usage of TiC or Ti_2B, new methods have to be developed to achieve a uniform distribution of nanoparticles on the surface in a stable state. Up to now, the aluminum powder was modified by adding an aqueous dispersion containing TiC nanoparticles and Polyethyleneimine (PEI) as a stabilizing agent. The stable dispersion was generated by using high power ultrasonic. For improved microstructure of the additive manufactured parts, TMC has enhanced the system for inoculating particles. Nanoparticles are added to an ethanolic system with a mixture of polymers as a stabilizing agent. The formed dispersion is sprayed on the aluminum particles and the resulting slurry is dried subsequently under vacuum for 16 hours. An exemplarily inoculated aluminum particle is given in figure 1.

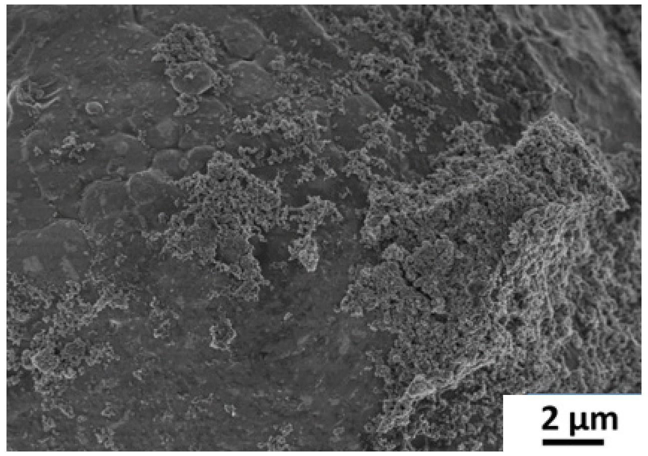

FIGURE 1 FE-SEM image of surface inoculated aluminum particle

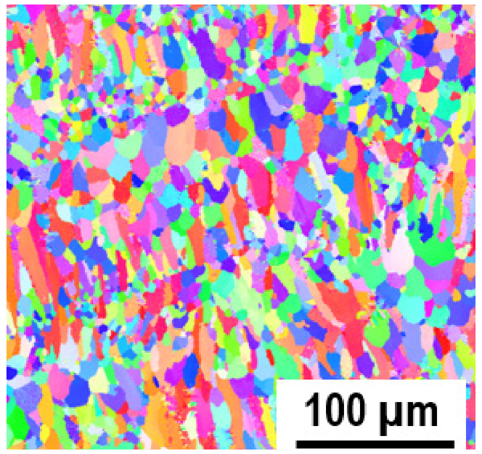

FIGURE 2 EBSD image of sample, made of blended, TiC modified EN AW-7075 powder

Results

Using a ring shear cell for powder characterizing unveiled a flowability enhancement for nanoparticle modified aluminum powder. The growing distances between each single aluminum particle of few nanometers cause the reduction of Van der Waals forces. A good flowable powder ensures a good coating during LBM. All samples made of powder which was modified with the aqueous method, exhibit cracks. The variation of LBM parameters did not lead to the envisaged objective of pore and crack reduction. However, grain refinement occurred and was proven with electron backscatter diffraction (EBSD). The decreased grain size and nearly globular grains at the edge of the melt pool are shown in figure 2. Therefore, different exposure strategies were investigated. For a second exposure during LBM, a reduction of crack size at the slight change of chemical composition was determined. With a consecutive heat treatment, hardness could be increased by 19 % to 20 % towards to specimens in additively manufactured as-build condition. Nevertheless, no significant improvement in crack reduction has been achieved. Moreover, it was only possible to investigate the combination of aluminum alloys with TiC.

The successful inoculation was proven for the aqueous and ethanolic dispersion by FE-SEM. The surface chemistry of the modified particles was further investigated by XPS. Upon the aqueous surface inoculation process, the natural passive layer of aluminum is thickened based on reactions with the aqueous dispersion. Due to the alkaline pH of the dispersion, the process of particle adsorption leads to an etching of the natural aluminum oxide coating. The natural aluminum hydroxides are then reconverted to oxide layers with adhering the nanoparticles during the drying process. For the ethanolic surface inoculation procedure, no oxide layer was detectable.

After substituting the aqueous procedure by an ethanol-based process to decorate the basic powders, new samples have been manufactured utilizing LBM. With the new powder, still hot cracks appeared. Independent of the amount of exposure and variation of layer thickness, the desired objective is not met. Currently, an extensive analysis of the correlation between parameter variation and both, pores and crack occurrence is performed. It seems that a reduction of cracks is possible but only with simultaneous increasing of numbers of pores.

Outlook

Due to the challenges of wet-chemical procedure for powder decoration, a mechanical method is taken into account: using a roller mill offers new possibilities. The ratio of initial state powder and grain refiner can be set easily. Moreover, almost unlimited powder combinations are possible. First investigations show the efficiency of mechanical decorating. Nevertheless, other procedures shall be examined concerning the applicability for powder decoration.

[1] Heiland, S.; Vieth, P.; Zhuravlev, E.; Hoyer, K.-P.; Grundmeier, G.; Schaper, M.; Keßler: Surface Inoculation of Aluminum Powders for Additive Manufacturing guided by Differential Fast Scanning Calorimetry. Lecture at LightMAT; Manchester, 6 11 2019.