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Influence of TiC-nanoparticles in Laser Metal Deposition of EN AW-7075

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Abstract

EN AW-7075 (AlZn5,5MgCu) is a high strength aluminum alloy for aerospace applications suffering from poor weldability due to solidification cracking susceptibility. In this study, crack free Laser Metal Deposition (LMD) of EN AW-7075 is enabled by adding up to 1 vol% of TiC-nanoparticles (35-55 nm) to the powder feedstock. It was found that nanoparticles are successfully incorporated into the melt pool where they provide grain refinement due to inoculation and thereby eliminate hot cracking.
Moreover, the addition of nanoparticles enhances the absorption of the laser wavelength in the powder (as measured with an Ulbricht sphere). It was found that the coupling efficiency of the processing laser beam was increased for the alloy to a small extent and no challenges due to reduced flowability occurred.

Keywords: Laser Metal Deposition; grain refinement; hot cracking; nanoparticles

1. Introduction

High-strength 7xxx-series aluminum alloys have suitable properties for example as lightweight structures in aerospace and large diameter bearing systems for wind turbines, where a high specific strength is important (Langebeck et al. 2020; Martin et al. 2017). EN AW-7075 (AlZn5,5MgCu) can have a tensile strength exceeding 500 MPa, enabling weight savings compared with steel components. However, these benefits come at the cost of poor weldability. The wide solidification range due to high zinc, magnesium and copper content promotes micro segregation of copper and zinc rich eutectics, hot cracking and liquation cracking during welding processes. Grain refinement can reduce hot cracks by reduction of size and orientation of low melting eutectics (Martin et al. 2017; Bai et al. 2016). Nanoparticles and especially TiC-nanoparticles have already shown grain refining properties in additive manufacturing of aluminum and steel, but the influence on process has not been fully addressed in the literature (Martin et al. 2017; Karimi et al. 2024; Zhai et al. 2022). There may be influences for example on absorption of the powder and flowability due to properties of TiC-nanoparticles. It was investigated whether they reach the melt pool and act as grain refiners at concentrations that do not pose challenges for applicants.

Previous works which addressed flowability with very smooth micro sized powders where nanoparticles and their agglomerates protrude over the roughness peaks has shown an improvement in flowability or a reduction in adhesive strength with a low influence of the chemical properties. Here the nanoparticles decide on the radius of the roughness contacting the neighboring particle and the distance to the neighboring particle. Due to the influence of stirring, these effects varied in intensity due to agglomeration, but there was no reduction in flowability or an increase in the adhesive force (Meyer and Zimmermann 2004; Gärtner et al. 2021). In contrast to this the flowability of a less spherical powder with satellites decreased with increasing loads of nanoparticles (Hentschel et al. 2024).

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2. Setup and Experimental Procedure

The substrate with a with a length and width of 60 mm and a thickness of 15 mm is made of the same material as the deposited sample. The powders EN AW-7075 (-106 µm +45 µm) supplied by ECKA Granules Germany GmbH and TiC (35 nm -55 nm) supplied by NANOGRAFI Nano Teknoloji A.S. were used.

Table 1. Chemical composition of the powder in weight %

Alloy	Zn	Mg	Cu	Cr	Mn	Si	Fe	Al
EN AW-7075	5.5	2.5	1.6	0.23	0.01	0.05	0.07	bal.

Mixtures of EN AW-7075 with 1 vol% TiC were produced by stirring for 5 min. The absorption, flowability and surface of the powders and their blends were characterized by following methods. The flowability of the powders was characterised based on standard EN ISO 4490:2008 whereby 50 g of the powder was passed through the Hall flowmeter with an opening of 2.5 mm and the time was measured. The absorption of the powders was measured by an Ulbricht-sphere with a wavelength of 1080 nm on a thick layer of powder. Furthermore, the structure of the powders was studied by SEM Zeiss EVO MA 10.

Nanoparticle concentrations lower than 1 vol% were reached by feeding EN AW-7075 powder without TiC and EN AW-7075 powder with 1 vol% in parallel in varying ratios through a powder blender. The blend was then deposited on the sample. Fig.1. shows the setup used for mixing the powder.

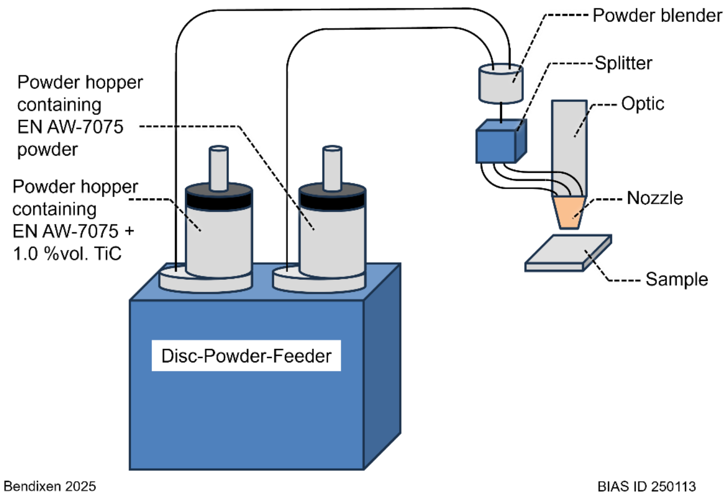

Fig. 1. Setup for powder mixing with a priorly mixed powder

The LMD experiments were conducted using a LUNOVU 835 LMD system equipped with a BLC CNS 37-25 nozzle. The setup included disc-powder-feeder and a vibration-powder-feeder. A Laserline LDF 4000-40 diode laser with wavelengths in range of 960 to 1040 nm, was used in combination with a Laserline OTZ-5 zoom optic and a 400 µm laser light cable. The laser spot exhibited a top-hat power intensity profile. The process parameters are shown Table 2.

Table 2. Process parameters

Process parameter	EN AW-7075
Laser power	1500 W
Travelling speed	400 mm/min
Powder feed rate	9 g/min
Shielding gas volume flow	8 l/min
Carrier gas volume flow	5 l/min
Laser spot diameter	3 mm

The coupling efficiency for the LMD of EN AW-7075 with and without 1 vol% TiC is measured. As reference, samples without addition of powder were remelted. For the aluminum alloy five layers high hollow cylinders are deposited with a layer height of 0.65 mm. The radius of the samples is 20 mm. This was done with the setup shown in Fig. 2.

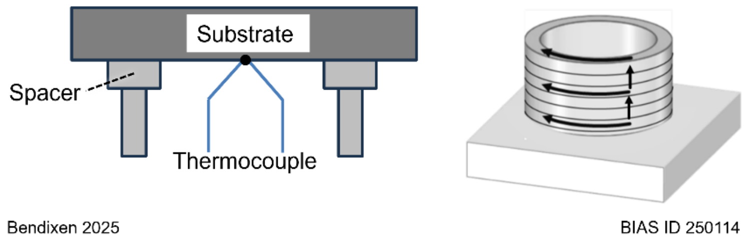

Fig. 2. Setup for the measurement of the coupling efficiency

In the setup the hollow cylinders were deposited centered on the substrate. At the bottom of the substrate thermocouples are mounted. By the spacers the heat conduction to machine bed is reduced. The measured temperature change ΔT is used to calculate coupling efficiency η_A by knowing the mass m, heat capacity c, laser power P_L and laser on time t.

$$\eta_A = \frac{m\ c\ \Delta T}{P_L\ t} \tag{1}$$

Metallographic cross sections were prepared and etched following Barker to measure the grain size. The influence of the addition of TiC on the grain size in EN AW-7075 was measured for concentrations lower than 1 vol%.

Results and Discussion

In the EN AW-7075 powder the absorption increased from 82.0 % ± 0.7 % to 93.0 % ± 0.6 % by addition of TiC. As an example in visible range Fig,3. (a)-(b) shows how the powder mixture with TiC appears darker than the pure EN AW-7075 powder. For comparison the TiC powder is shown in Fig 3. (c) which absorbs nearly all light in visible rage and at measuring wavelength.

The powder particles are roundish with some larger satellites with a size up to 20 µm and many smaller satellites up to 5 µm. This is shown as an example in Fig. 3. (d) for the EN AW-7075 powder. Fig. 3. (e) depicts the surface of a powder particle of the aluminum alloy covered with agglomerates of TiC is shown. The rough appearance of the surface is due to the covering of small agglomerates of TiC. The light-colored areas in the center and on the bottom right of the image are larger agglomerates. The high degree of coverage of the nanoparticles can explain increased absorption even with a relatively low addition of 1 vol% TiC. The agglomerates are much smaller than the unevenness on the particle or than the satellites shown in Fig. 3. (d). Fig. 3. (f) shows a smaller agglomerate of TiC on the top left and some particles on the right.

As result of flowability tests, the throughput time of the powders without the addition of TiC was the shortest. The EN AW-7075 powder had a throughput time of 80.1 s ± 0.6 s and the addition of 1 vol% TiC increased the time to 87 s ± 1.5 s. The pure TiC powder had no flowability according to the standard. In consequence for further experiments the premixtures with 1 vol% TiC were chosen, because a flowability promises the ability to be fed in LMD process.

The slight deterioration in flowability with the addition of TiC nanoparticles matches with the observation in the work of (Hentschel et al. 2024). Moreover, there occurrence of non-spherical particles and satellites on the surface of the particles is higher. The reason could be that the uneven surface of the microparticles reduces the influence of the nanoparticles as spacers with a low contact area and radius because a high percentage of the nanoparticles and their agglomerates did not protrude over the satellites on the surface on the powder.

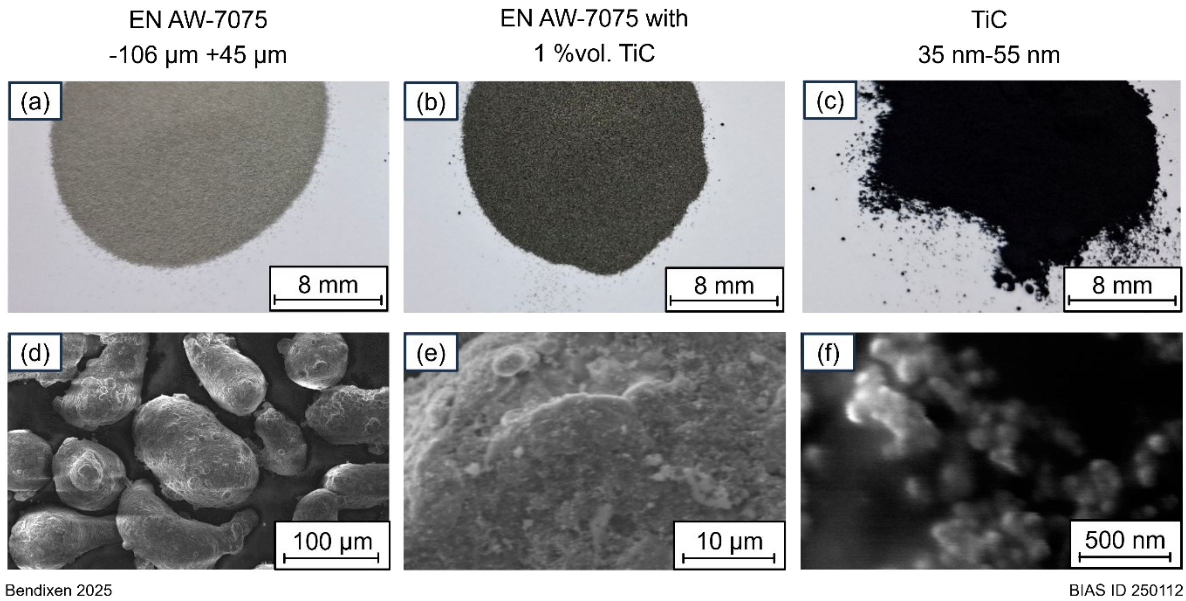

Fig. 4. (a) - (c) images of piles of the different powders which looks darker with increasing TiC content; (d) - (f) SEM images of the powders

The coupling efficiency (Fig.4.) for substrate remelting of EN AW-7075 was 34.6 % ± 0.7 %. The value increased to 47.4 % ± 0.3 % when EN AW-7075 is deposited and is further increased to 51.4 % ± 0.3 % when TiC is added the deposited powder. The increase of coupling efficiency due to the addition of the metal powder fits to the expectation, as the powder also absorbs part of the light and a significantly more uneven surface is created. (Kaplan 2014). Also, the addition of TiC increased the coupling efficiency when EN AW-7075 is deposited. This may be explained due to the good absorption, high melting point and surface coverage of the TiC-nanoparticles on the aluminum particles.

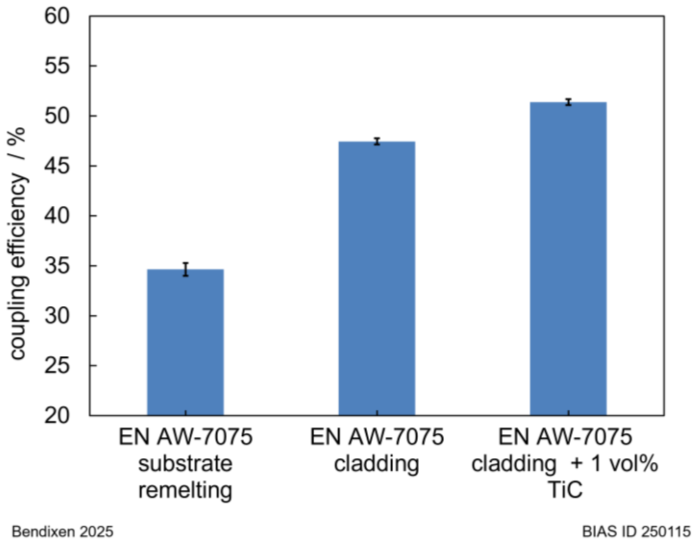

Fig. 3. Coupling efficiency for the investigated material combination

Without the addition of TiC, the EN AW-7075 samples have a median grain size of 267 µm and the microstructure is highly orientated. As shown in Fig. 5. (a), a hot crack can also be seen in the center. Hot cracks are common in samples without TiC. With an increasing addition of TiC (Fig. 5. (b) – (d)), the grain size decreases and from an addition of 0.75 vol% a homogeneous and fully equiaxed microstructure occurs. The smallest grain size of 12 µm, which is very small for an aluminum sample produced using LMD, is achieved with the highest addition of 1 vol% TiC (Fig. 5. (e)).

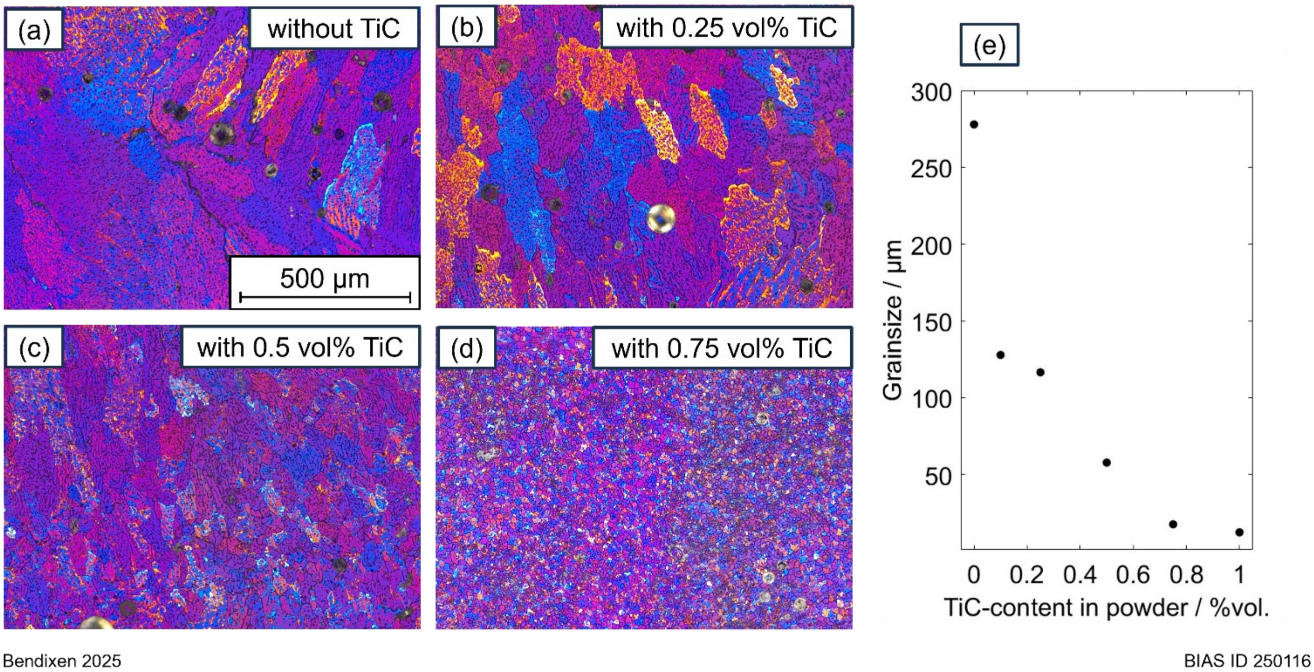

Fig. 5. Influence of the addition of TiC-nanoparticles on the grain size

With the highest addition of TiC hollow cylinders could be built up without suffering from hot cracking, as shown in Fig.6. In the first layers there occurred pores but in higher layers there are nearly no imperfections

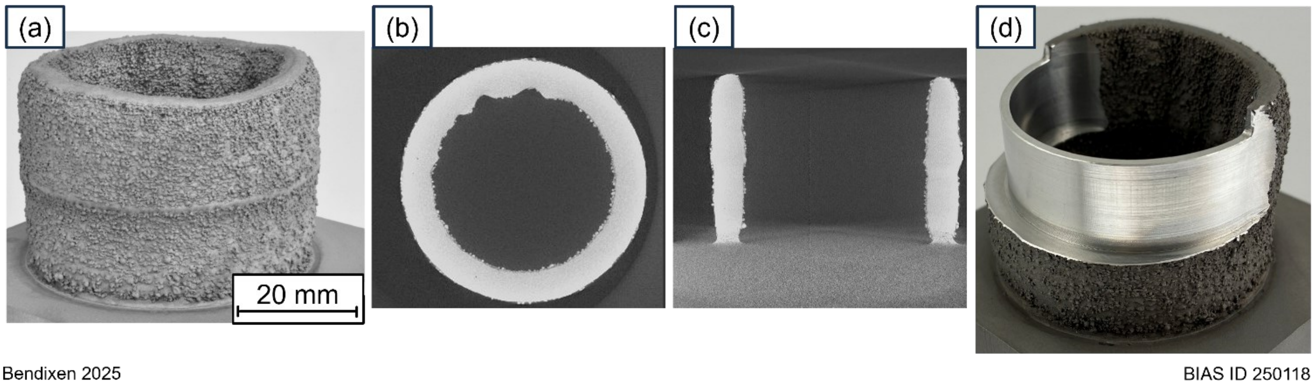

Fig. 6. Hollow cylinder build up out of EN AW-7075 with 1 vol% TiC: (a) as printed; (b) plane out of X-ray CT-scan parallel to substrate plane; (c) plane out of X-ray CT-scan intersecting substrate plane at a 90° angle; (d) partially milled

3. Conclusion

- EN AW-7075 was blended with up to 1 vol% TiC-nanoparticles and the influence on the absorption of the powder, the coupling efficiency and the powder flowability was focused.
- The absorption of the powders is increased by the addition of TiC-nanoparticles and the coupling efficiency is increased.
- The grain size is decreased from 267 µm to 12 µm by addition of 1 vol% TiC. This shows that the TiC-nanoparticles are successfully incorporated into the melt pool where they provide grain refinement due to inoculation and thereby eliminate hot cracking.
- With up to 1 vol% TiC samples of high integrity were produced without challenges regarding flowability and coupling efficiency.

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