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Details see *https://doi.org/10.1016/j.jmatprotec.2021.117357*

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In-situ formation of particle reinforced Al matrix composites by laser melting + remelting strategy in laser powder bed fusion of Fe_2O_3/AlSi12 powder mixture

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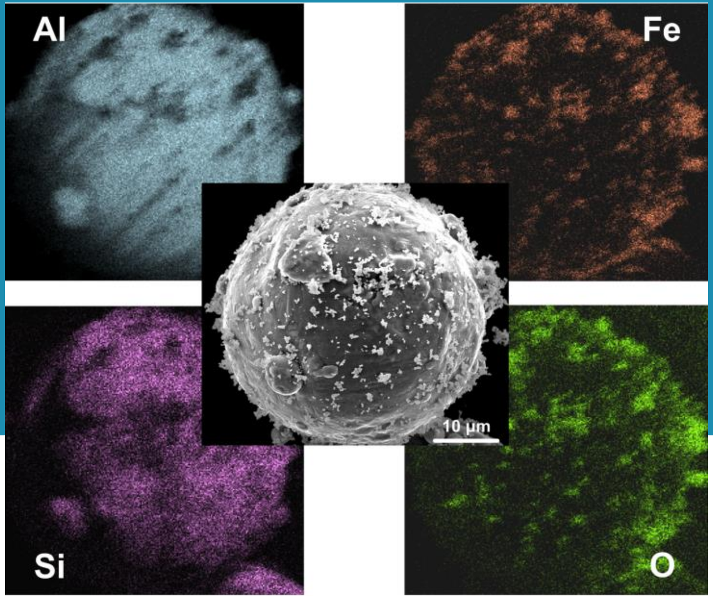


International Conference on Additive Manufacturing Materials for Production | Barcelona, Spain, 16 Dec. 2021

Outline

- **Introduction**
- **Materials and methods**
- **Results and discussion**

  Densification | Top surface & cross section morphology

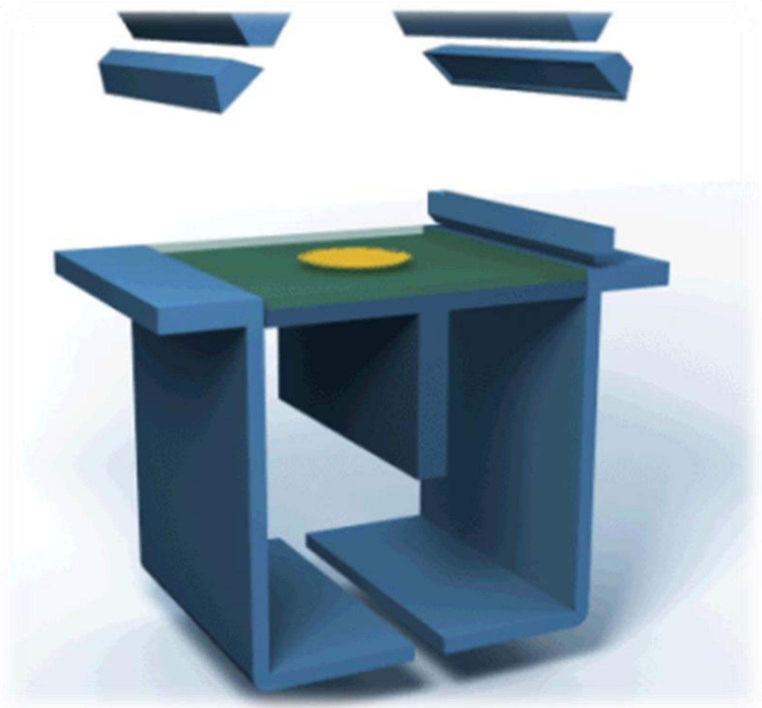
  Microstructure & Phase | Microstructural evolution with phase analysis

  Mechanical performance | Tensile property

- **Conclusions**

Introduction | LPBF

- **LPBF** is a metal additive manufacturing technology that uses *a bed of powder* with *laser heat source* to create metal parts in a *layer-by-layer manner*

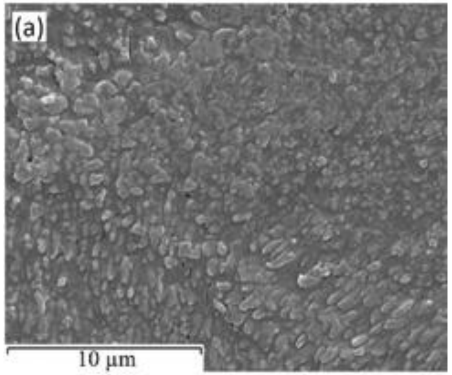


Introduction | Advantages

- **In-situ formation** of particle reinforced Aluminium matrix composites (PRAMCs) by LPBF of **Fe_2O_3/AlSi12 powder mixture** is a promising strategy to strengthen Al-based alloys

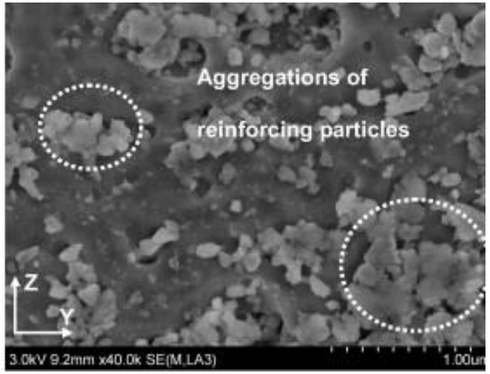
(1) 'In-situ' mechanism creates stronger interfacial bonding & better distribution of fine reinforcements, compared to the 'ex-situ' mechanism

In-situ



Uniform distribution of fine Al_2O_3 and $FeAl_2O_4$ particles in LPBF-fabricated parts from Fe_2O_3/Al powder mixture
[Adv. Eng. Mater. 21(2019) 1801244]

Ex-situ

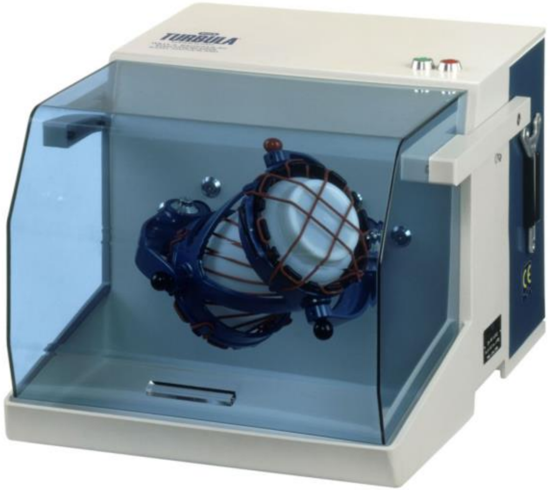


TiC aggregations in LPBF-fabricated TiC/Al composites
[Mater. Des. 82(2015) 46-55]

Introduction | Advantages

(2) Fe_2O_3/AlSi12 powder mixture is easy to prepare, compared to precursor powders

Direct mechanical mixing to prepare Fe_2O_3/AlSi12 powder mixture



3D shaker-mixer

Multiple procedures to prepare precursor powders

e.g., Preparation of graphene oxide coated AlSi10Mg powder to prepare:

Step 1, mechanical stirring and ultrasonication of AlSi10Mg in water in an ice-water bath for 1h;

Step 2, adding graphene oxide drop-by-drop into the AlSi10Mg suspension under mechanical stirring;

Step 3, filtration and vacuum drying at 298 K for 24h.

[Mater. Charact. 170(2020) 110678]

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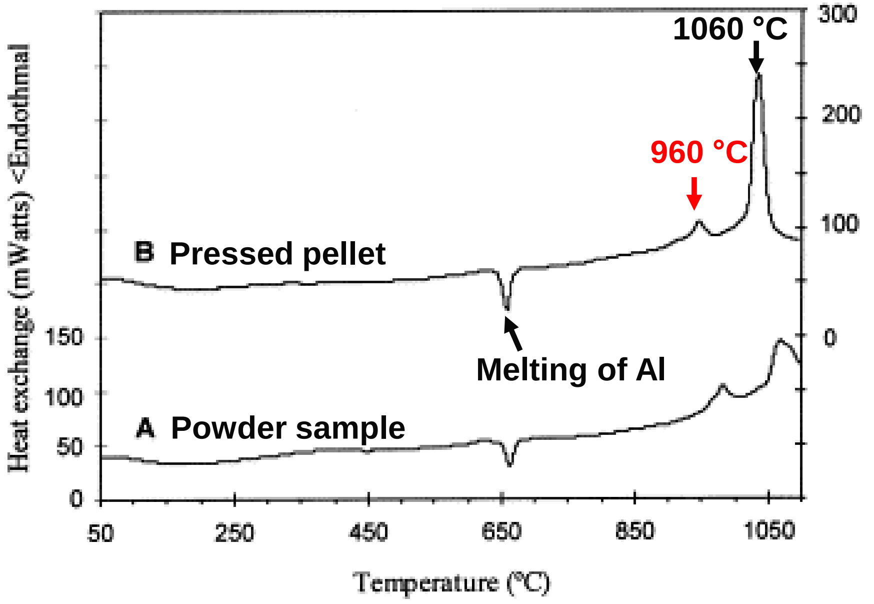
Introduction | Advantages

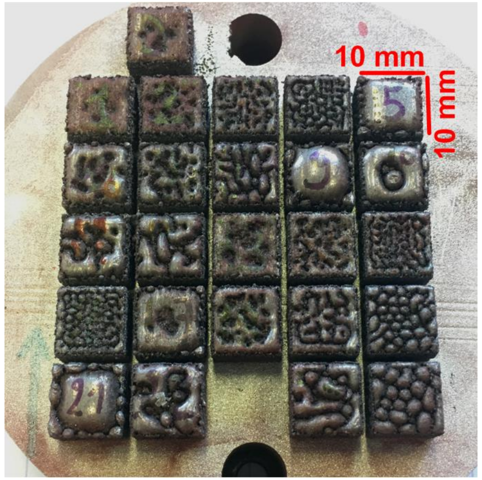
(3) In-situ Fe_2O_3/Al reaction is easy to initiate at 960 °C



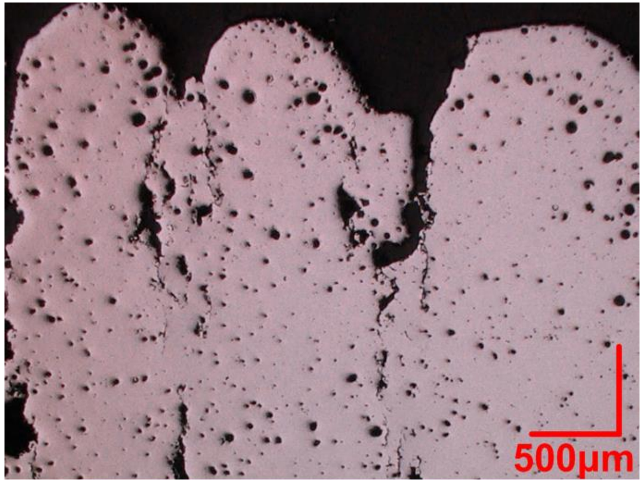
Differential thermal analysis of mixed Fe_2O_3/Al samples
[Scripta Mater. 41(1999) 541-548]

Introduction | Challenge

- By-product O_2 reacts with Al, forming Al_2O_3 film that restricts the metal liquid's spreading and impact the densification
- **Objective**: densify the LPBF parts from the Fe_2O_3/AlSi12 powder mixture



Without laser remelting: Top surface of LPBF parts from the Fe_2O_3/AlSi12 powder mixture



Without laser remelting: Cross section of LPBF parts from the Fe_2O_3/AlSi12 powder mixture

Materials and methods | Powder preparation

- Homogeneous 5wt%Fe_2O_3/AlSi12 powder mixture was obtained by mechanical mixing for 10 hours

AlSi12

| Gas-atomized, spherical

| Particle size < 45 µm

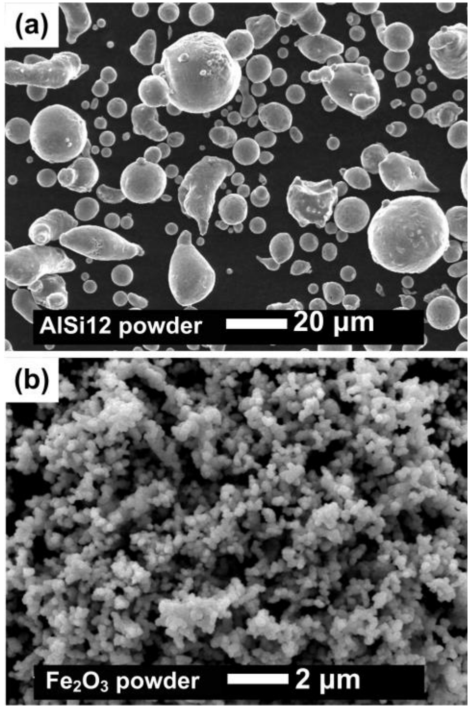
| Average size = 25 µm

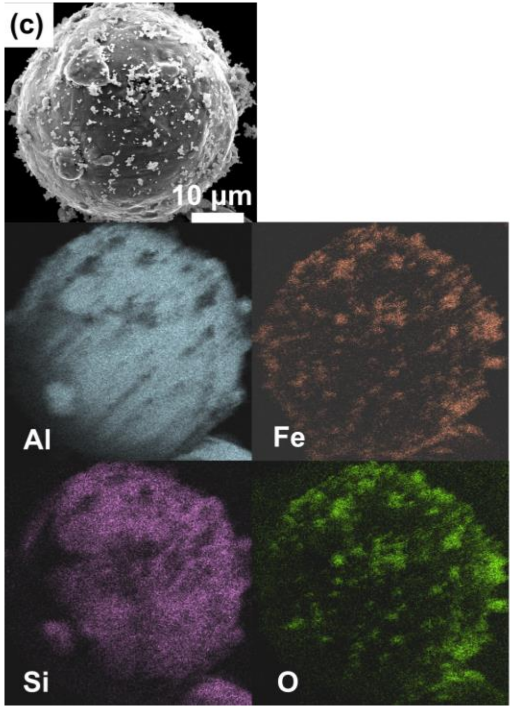
Fe_2O_3

| Spherical

| Average size = 200 nm

| Purity = 99.8%



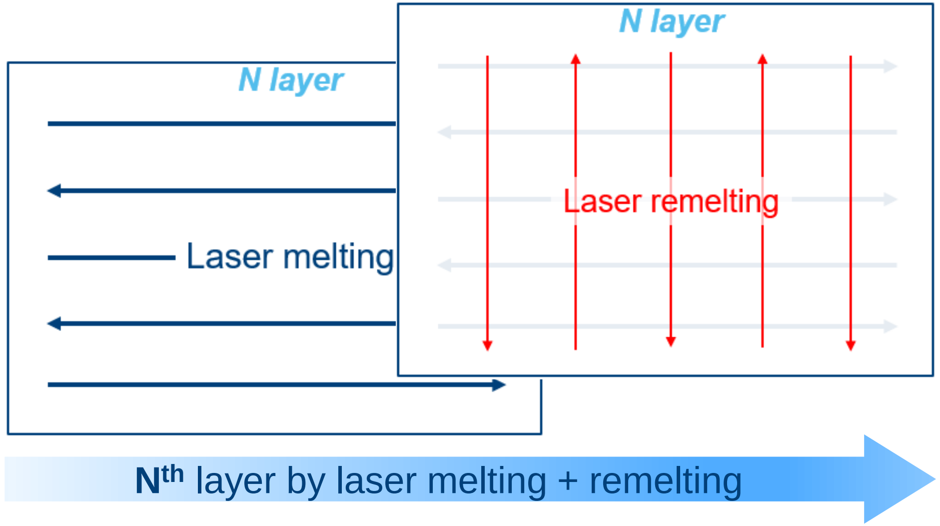


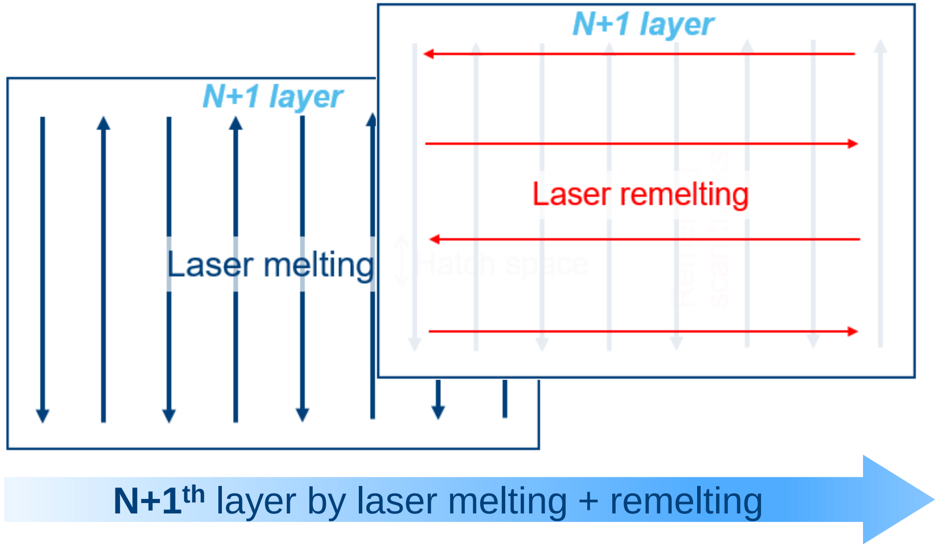
Powder mixture

| Fine Fe_2O_3 is well assembled on the surface of AlSi12

Materials and methods | Laser scan process

- A consecutive **high-energy melting scan** and **low-energy remelting scan** was used in the LPBF process





Materials and methods | Laser parameters

- **Melting process optimization**

  Variable $E_{melting}$: 20, 25, 30, 35 J/mm^2

  Fixed $E_{remelting}$: 4.17 J/mm^2

	P **(W)**	***v*** **(mm/s)**	***h*** **(μm)**	***t*** **(μm)**
Melting	100-175	100	50	30
Remelting	250	600	50	/

- **Remelting process optimization**

  Fixed $E_{melting}$: 35 J/mm^2

  Variable $E_{remelting}$: 2.92, 4.17, 4.29, 5.00 J/mm^2

	P **(W)**	***v*** **(mm/s)**	***h*** **(μm)**	***t*** **(μm)**
Melting	175	100	50	30
Remelting	210-350	600-1200	70-100	/

- **Comparison group**

  Same laser melting parameters but no remelting

Laser energy surface density (J/mm^2)

$E_{melting} = P/v \cdot h$

$E_{remelting} = P/v \cdot h$

P, laser power; ***v***, laser scan speed;
h, hatch space; ***t***, powder laser thickness

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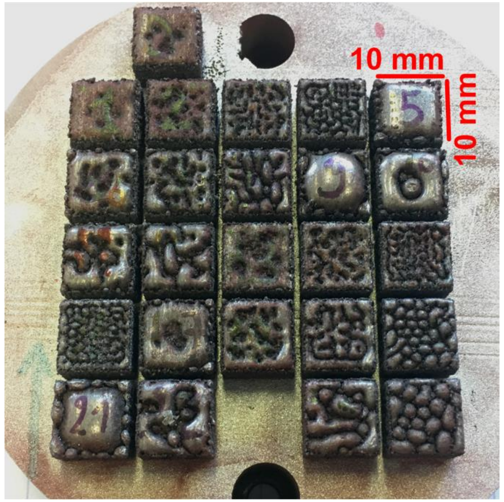
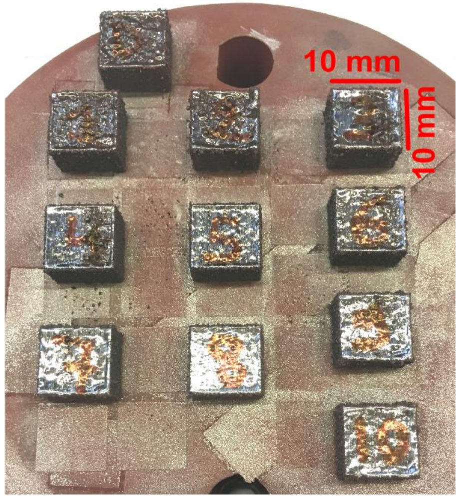
Materials and methods | Outcomes

- Big improvement of sample quality was achieved by laser melting + remelting

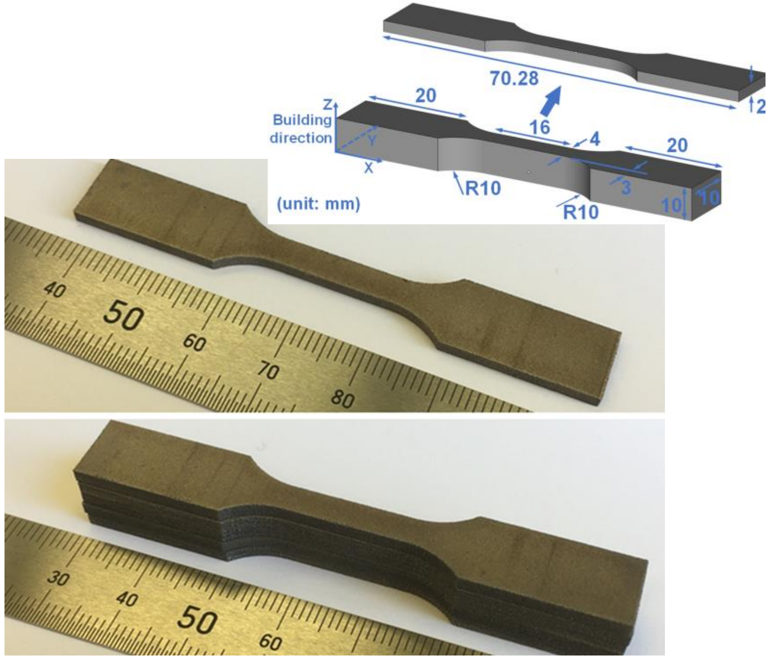


Only laser melting process

vs.



Laser melting + remelting process

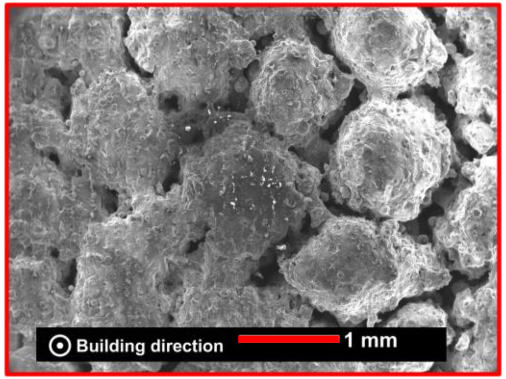


Tensile pieces by laser melting + remelting process

Densification | Top surface morphology

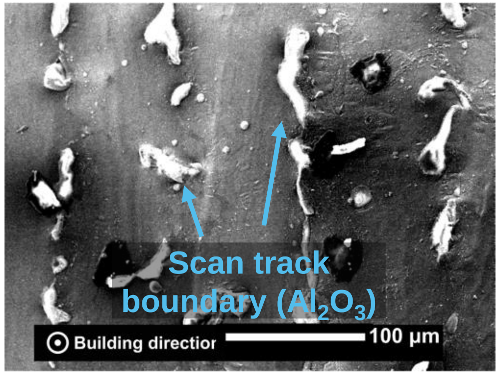
- Laser melting + remelting smoothened the top surface

$E_{melting}$ of 35 J/mm^2
No remelting!



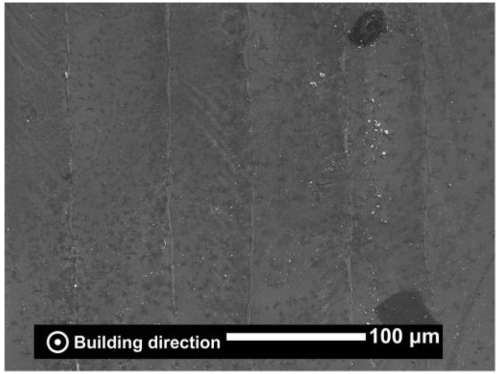
Melting process optimization

$E_{melting}$ of 35 J/mm^2
$E_{remelting}$ of 4.17 J/mm^2



Remelting process optimization

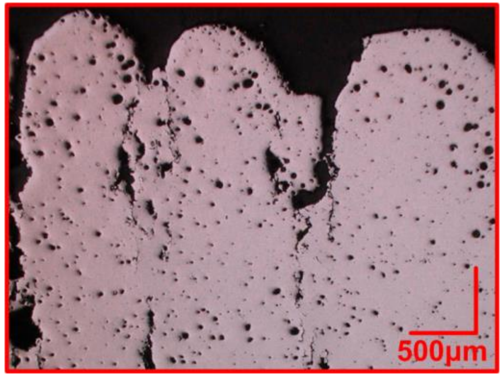
$E_{melting}$ of 35 J/mm^2
$E_{remelting}$ of 5.00 J/mm^2



Densification | Side surface morphology

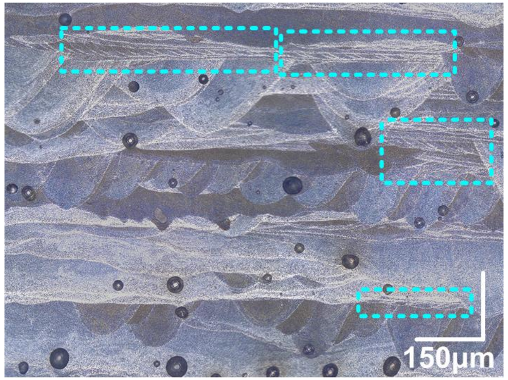
- Laser melting + remelting densified the sample by forming horizontally layered solidified structure

$E_{melting}$ of 35 J/mm^2
No remelting!



Melting process optimization

$E_{melting}$ of 35 J/mm^2
$E_{remelting}$ of 4.17 J/mm^2

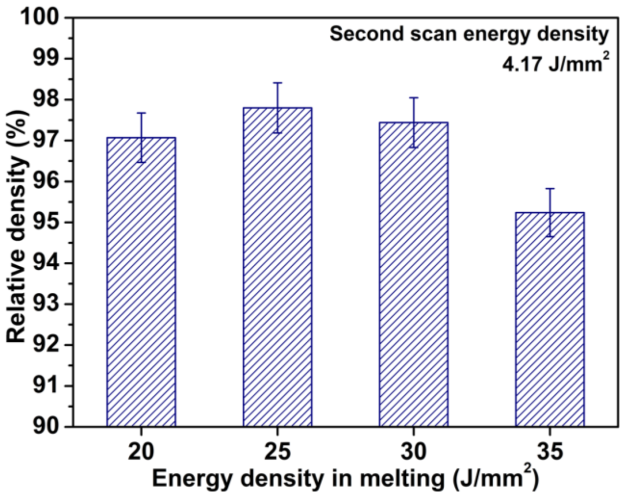


Remelting process optimization

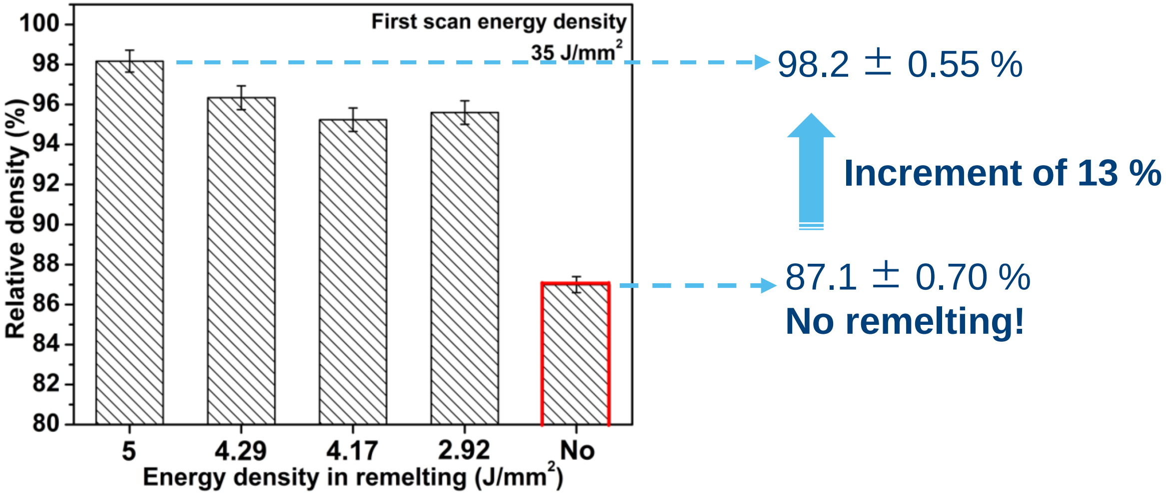
$E_{melting}$ of 35 J/mm^2
$E_{remelting}$ of 5.00 J/mm^2

Densification | Relative density

- Laser melting + remelting improved the relative density to **98.2 ± 0.55 %**



Melting process optimization



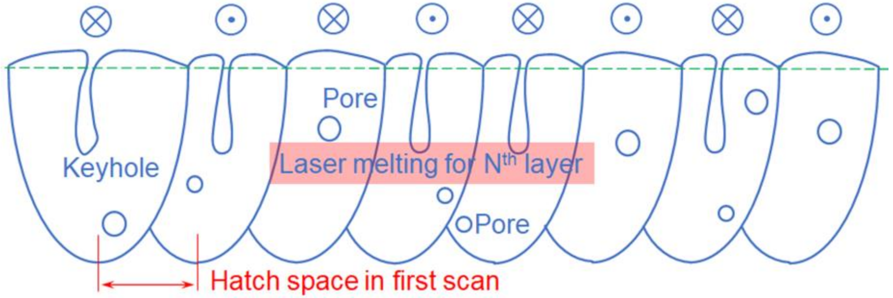
Remelting process optimization

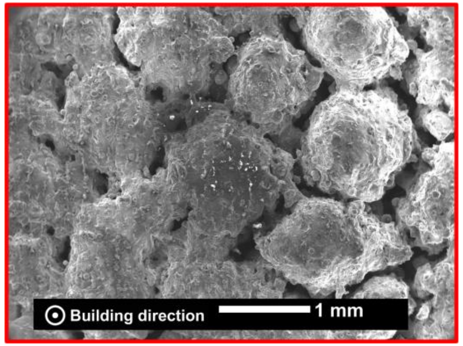
Densification | Mechanism behind densification

- Laser remelting smoothened the top surface and closed gaps layer by layer

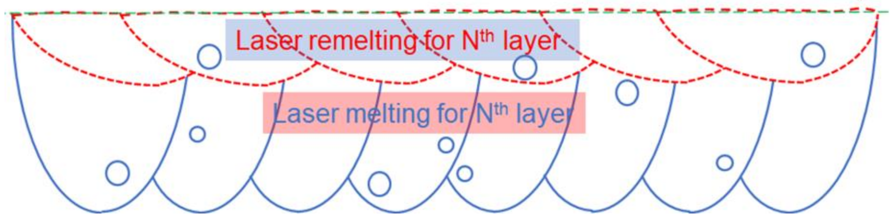
N^th layer

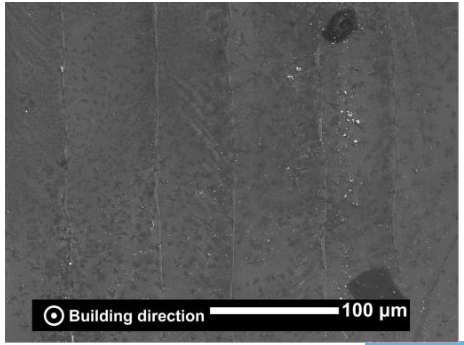
After laser melting scan
(O_2-, H_2- and keyhole- induced pores)





After laser remelting scan

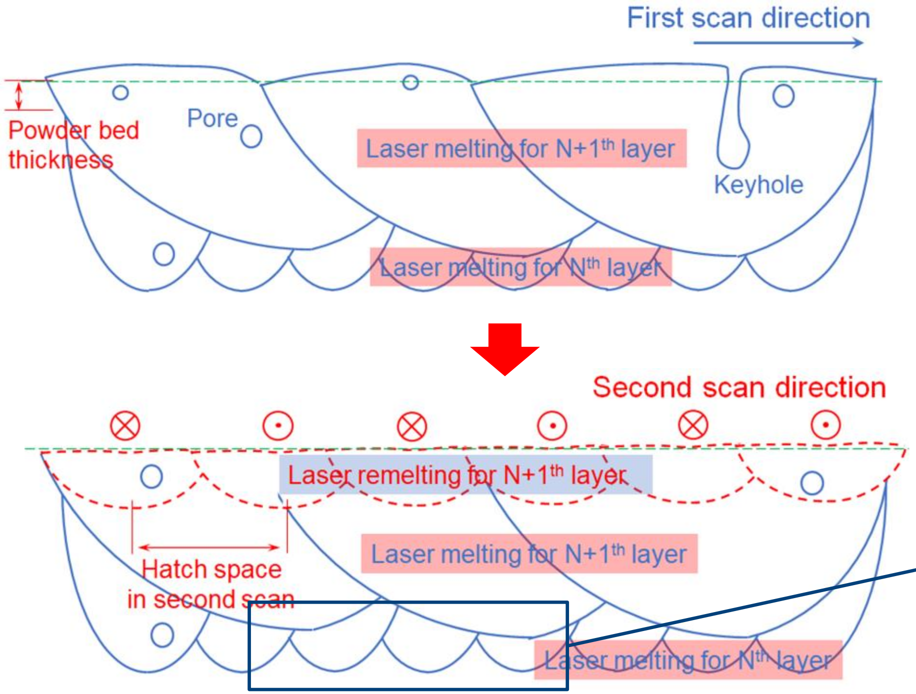


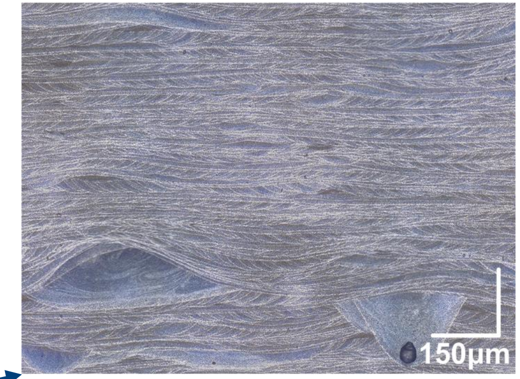


Densification | Mechanism behind densification

- Repeated laser melting + remelting produced fine horizontally layered solidified structure

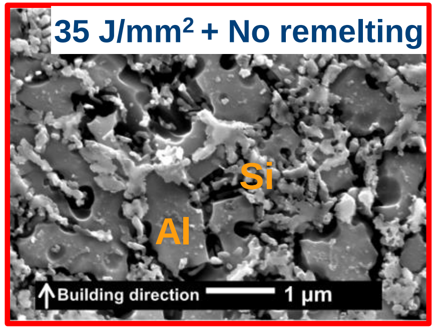
N+1th layer



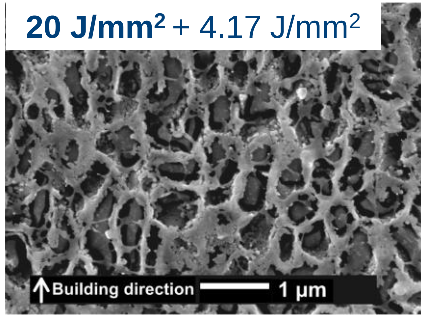


Microstructure & Phase | Microstructural evolution

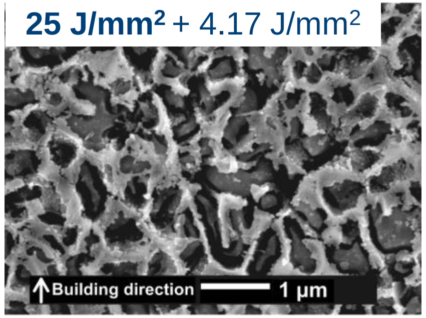
- Laser melting + remelting did not coarsen the microstructure



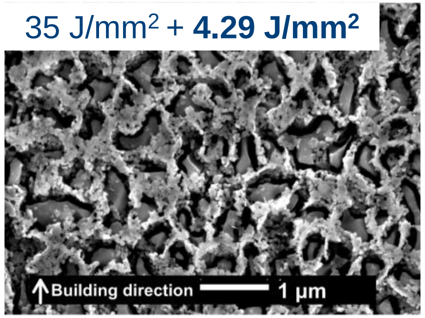
| Grain size
900 µm
| Hardness
130±3.1 HV



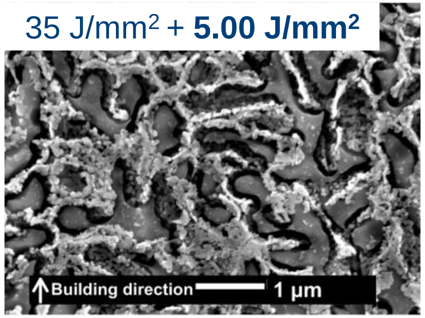
| Grain size
370 µm
| Hardness
144±3.7 HV



| Grain size
420-600 µm
| Hardness
138±2.5 HV



| Grain size
410 µm
| Hardness
144±1.4 HV



| Grain size
655-710 µm
| Hardness
136±2.2 HV

Increasing laser energy density

No remelting! **Melting** process optimization **Remelting** process optimization

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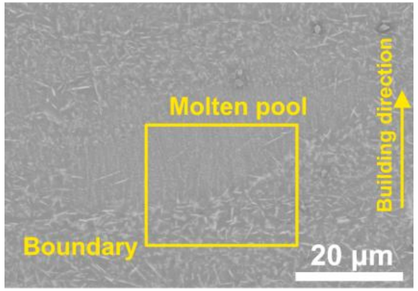
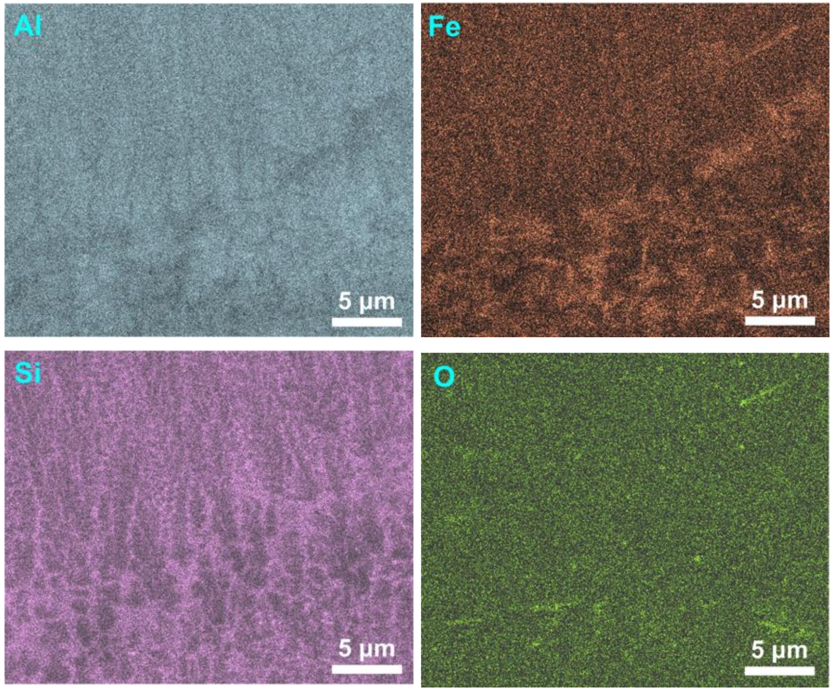
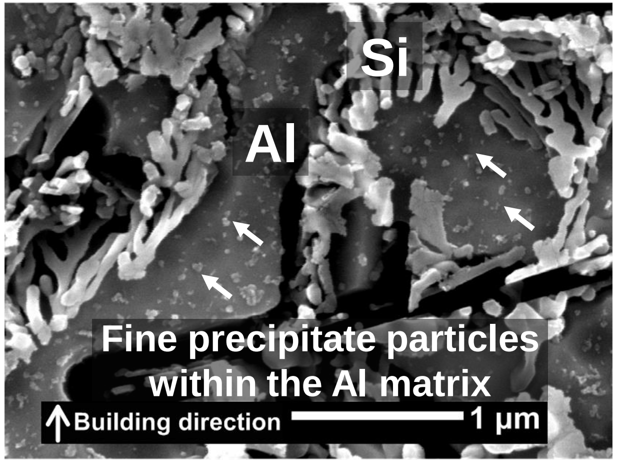
Microstructure & Phase | Precipitate distribution

- EDS maps confirm the distribution of precipitate-related elements (Al, Si, Fe and O) across the whole material







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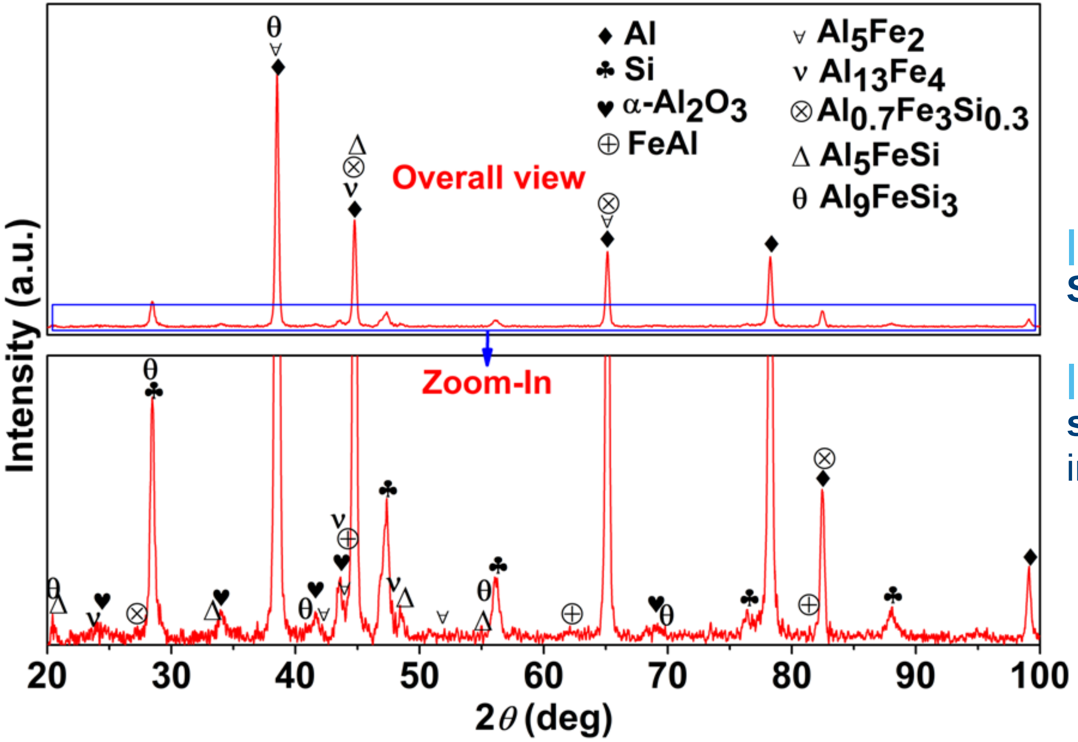
Microstructure & Phase | Phase evaluation

- Laser melting + remelting produced multiple precipitate phases

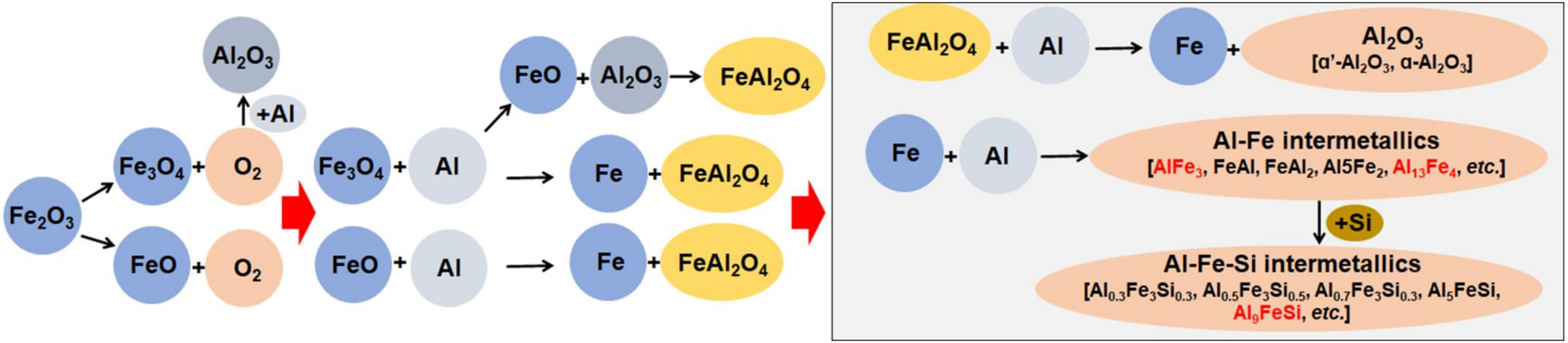


| Main phases of **Al matrix** and **Si particles**

| Minor phases of **Al_2O_3**, and **stable/ metastable Al-Fe(-Si)** intermetallics

Microstructure & Phase | Phase evaluation

- In-situ Fe_2O_3/Al reaction includes multiple steps, providing enough possibilities of forming precipitates [1,2]



[1] Thermochim. Acta 440 (2006), 129–131.
[2] Mater. Sci. Eng. A 465 (2007), 199–210.

Microstructure & Phase | Phase evaluation

- Rapid cooling process of LPBF froze intermediate metastable precipitates

Primary metastable phases

$AlFe_3$
$Al_{0.3}Fe_3Si_{0.7}$
α'-Al_2O_3

Intermediate metastable phases

$AlFe_3$
$FeAl_2$
Al_5FeSi
...

Final stable phases

$Al_{13}Fe_4$
Al_9FeSi_3
α-Al_2O_3

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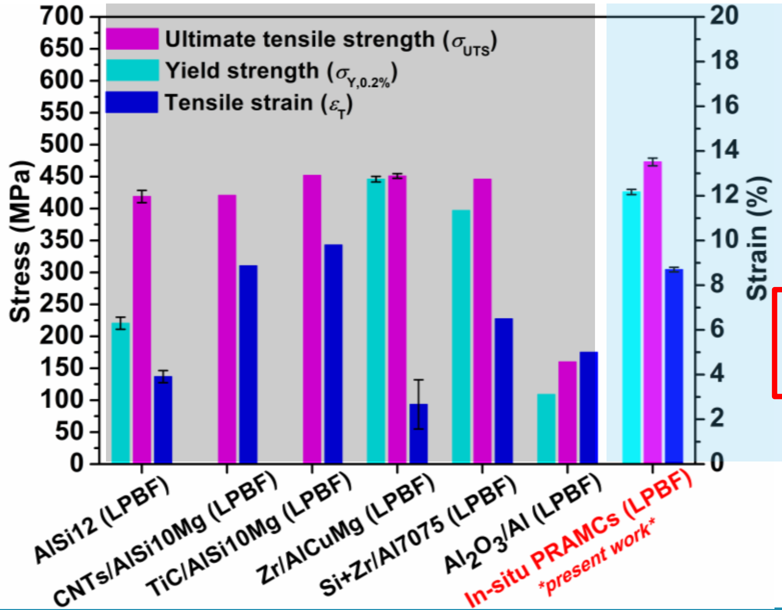
Mechanical performance | Tensile properties

- In-situ PRAMCs by LPBF have **comparable/ superior mechanical performance** to LPBF-fabricated Al-based composite counterparts/ cast AlSi12.

Al-based composite counterparts by LPBF

In-situ PRAMCs from Fe_2O_3/AlSi12 powder mixture by LPBF

Cast AlSi12 [1,2]

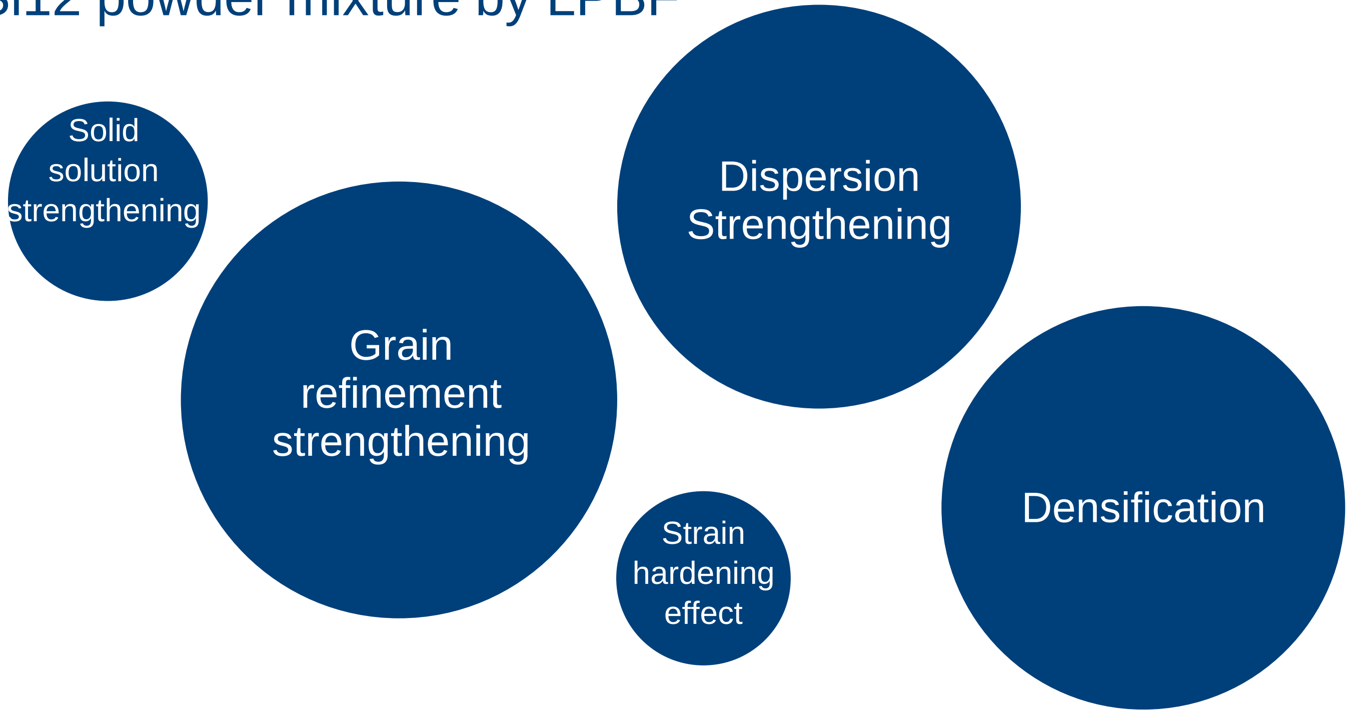


In-situ PRAMCs	Cast AlSi12
Ultimate tensile strength **473±6 Mpa**	Ultimate tensile strength **130-230 Mpa**
Yield strength **426±4 Mpa**	Yield strength **55-110 Mpa**
Tensile strain **8.7±0.2 %**	Tensile strain **8-9.5 %**

[1] J. Mater. Process. Technol. 221, 205–213.
[2] Mater. Sci. Eng. A 590, 153–160.

Mechanical performance | Strengthening mechanism

- Multiple contributions strengthened the in-situ PRAMCs made from the Fe_2O_3/AlSi12 powder mixture by LPBF



Conclusions

- The consecutive laser melting + remelting scan strategy can prepare **dense PRAMCs** from Fe_2O_3/AlSi12 powder mixture.
- The PRAMCs made from Fe_2O_3/AlSi12 powder mixture by LPBF achieve good mechanical properties.
- Laser melting + remelting could be practical to process other powder mixtures with severe porosity/ oxide-film formation potential

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For further details

- Qimin Shi, *et al.*, In-situ formation of particle reinforced Aluminium matrix composites by laser powder bed fusion of Fe_2O_3/AlSi12 powder mixture using laser melting/remelting strategy, *Journal of Materials Processing Technology* 299 (2022) 117357.

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In-situ formation of particle reinforced Aluminium matrix composites by laser powder bed fusion of Fe_2O_3/AlSi12 powder mixture using laser melting/remelting strategy

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Thank you for your attention

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University of Southampton, Faculty of Engineering and Physical Sciences, UK

Material characterization

- **OM**: Optical Microscope (Keyence VHX6000 microscope, Japan)
- **Etching**: Keller's reagent (95 mL H_2O, 2.5 mL HNO_3, 1.5 mL HCl and 1 mL HF for 60s)
- **XRD**: Seifert 3003 T/T X-Ray diffractometer, at 40 kV and 40 mA, step-scan type with a step width of 0.02◦ and exposure time of 1 s for each step, 2θ = 20-100º
- **SEM**: Scanning Electron Microscope (Philips XL30 FEG, Eindhoven, Netherlands)
- **EDS**: Energy Dispersive Spectrometer (EDS; EDAX Inc., USA)
- **Hardness**: FV-700 microhardness tester, load of 500 g during 15 s
- **Tensile**: Instron 4467 testing facility, tensile rate of 0.2 mm/min