

RADIKAL - Ressourcenschonende Werkstoffsubstitution durch additive & intelligente FeAl-Werkstoff-Konzepte für angepassten Leicht- und Funktionsbau



GEFÖRDERT VOM



Bundesministerium
für Bildung
und Forschung



Additive Manufacturing of Intermetallics: Microstructures and Mechanical Properties of Iron Aluminides Processed by SLM and LMD

M. Palm



- Iron aluminides and investigated Fe–Al–X alloys
- Microstructures
 - Misorientations within individual grains
 - Influence of preheating temperature, scan strategy, annealing
- Mechanical properties: yield strength, creep, ductility
- Chemically graded Fe–Al and Fe–Al/steel samples



- Fe-base materials with low density $5.7 - 6.7 \text{ g/cm}^3$ vs. 7.85 (steel)
- Excellent corrosion resistance (metal dusting, steam, molten salts...)
- High wear resistance
- Progress in increasing strength at high temperatures
- Limited ductility at ambient temperatures (0.5–1.5% elongation at RT)
- Cheap material (materials costs, no strategic elements & production)

⇒ **Possible replacement for Cr steels and Ni & Co base alloys**

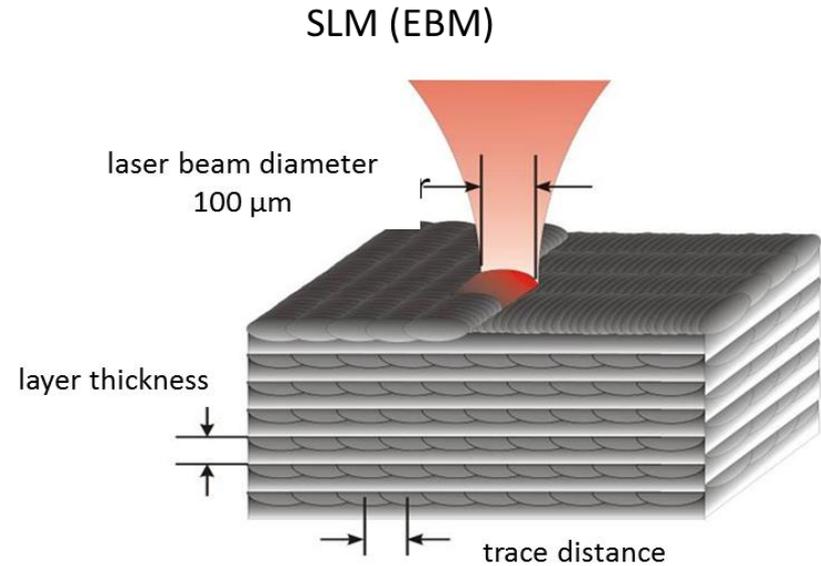
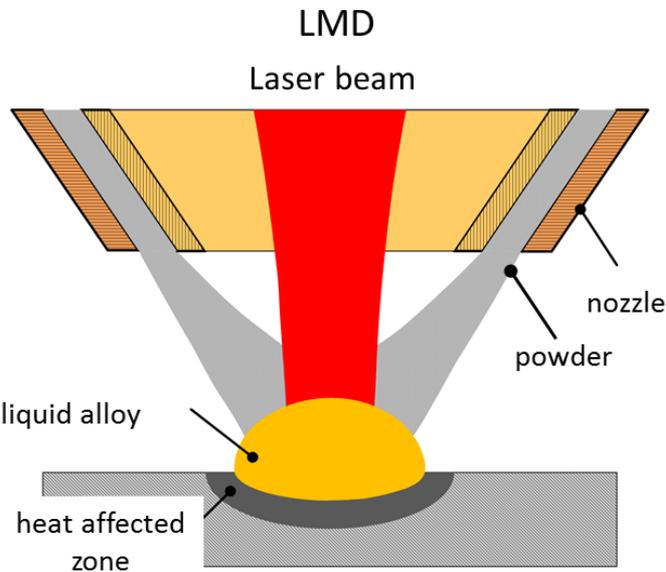
AM processing is of specific interest for the production of intermetallic parts because their high hardness and brittleness makes near net-shape production desirable.

Why additive manufacturing (AM) of Fe–Al?

- Alternative processing route to casting
- Near net-shape processing \Rightarrow minimum of machining
- High cooling rates \Rightarrow fine microstructure \Rightarrow higher ductility
- Highly exothermic reaction between Fe and Al
 - \Rightarrow build up from elemental powders ✓
- Possibility to build chemically graded steel/Fe–Al parts?

Employed AM processes

1. Laser Metal Deposition **LMD**
2. Selective Laser Melting **SLM**
3. (Electron Beam Melting **EBM**)



Build up on various preheated substrates: Fe, Fe–Al, 1.4301, 1.1730, P92...

Reaction zone between substrate and iron aluminide ≤ 1 mm

Investigated Fe–Al–X alloys

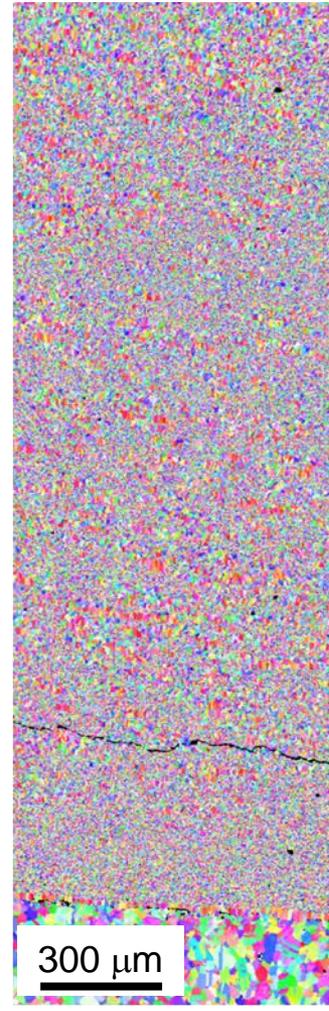
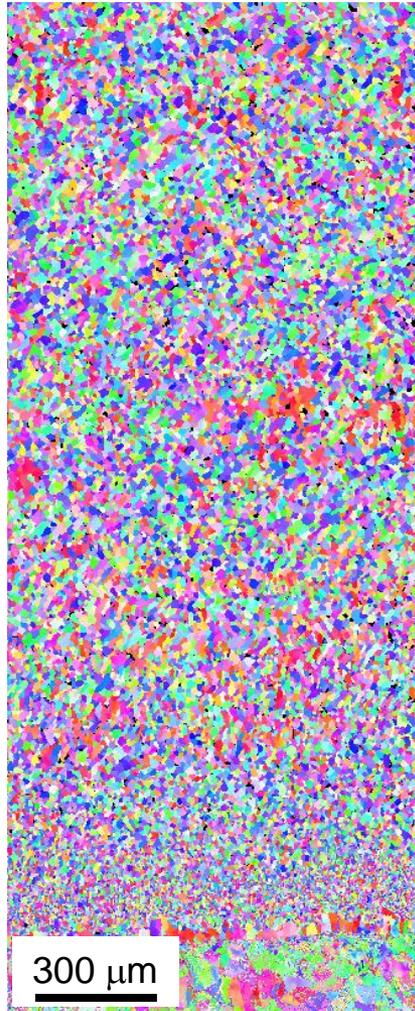
Fe–28Al
“Demo”

Fe–30Al–10Ti
Increase of $L2_1 \leftrightarrow B2$

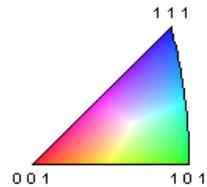
Fe–30Al–5Ti–0.7B
Precipitation of
borides

Fe–22Al–5Ti
Coherent $A2 + L2_1$

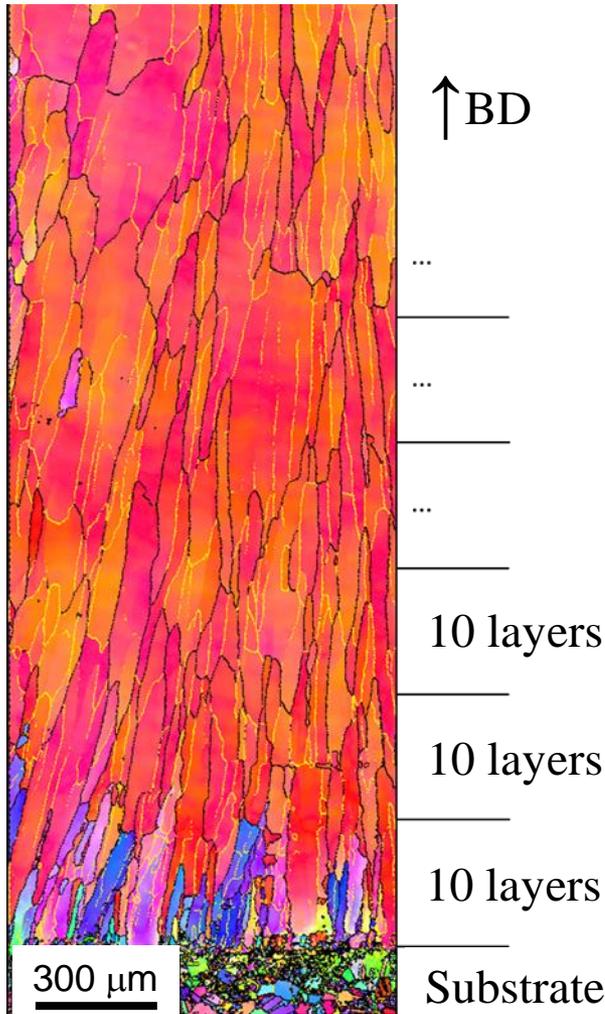
EBSD (SLM)



↑BD



Fe-28Al “Demo”

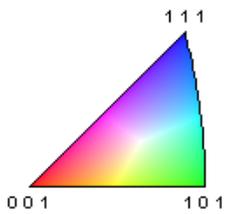


Defect free samples by preheating
at 200 °C (LMD) and 600 °C (SLM)

Compositions SLM: 27.2 ± 0.3 at.% Al
 LMD: 27.8 ± 0.3 at.% Al
 Powder: 28.3 at.% Al

Elongated grains up to mm (as cast several 100 μm)

Growth preferentially in [001] direction
(direction of the highest heat flow in α-Fe,Al)



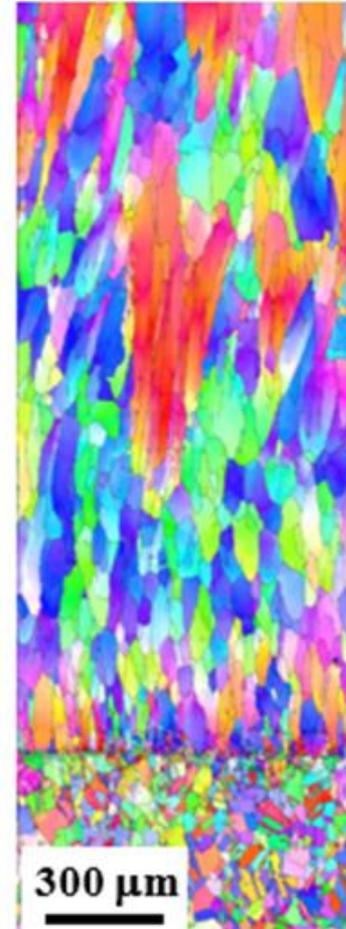
Without preheating



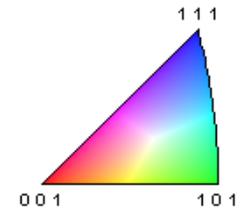
Preheating 200 °C



Preheating 600 °C



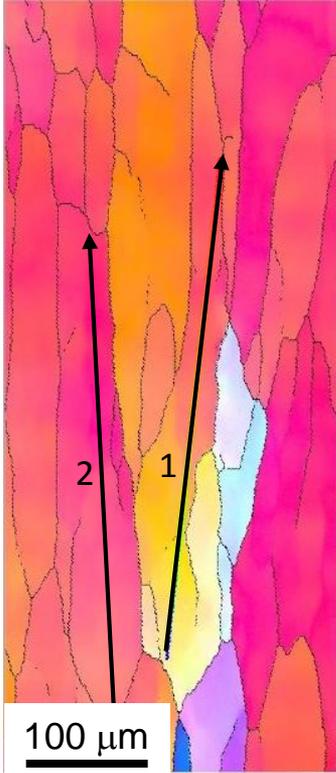
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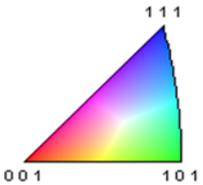
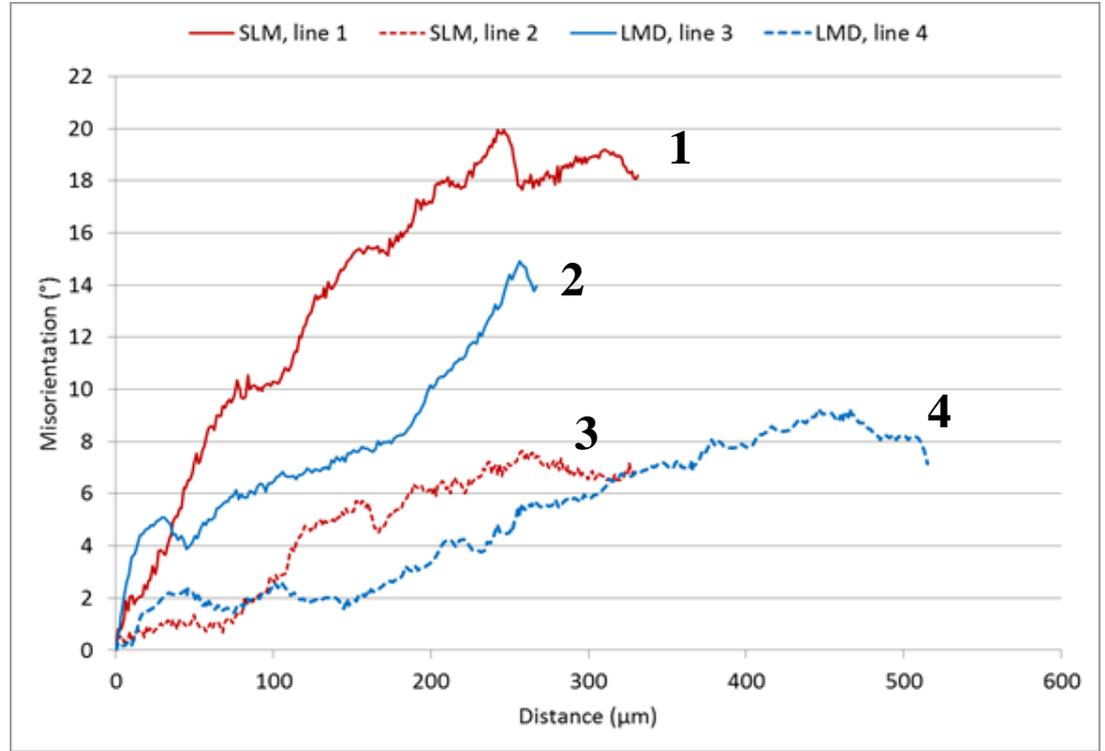
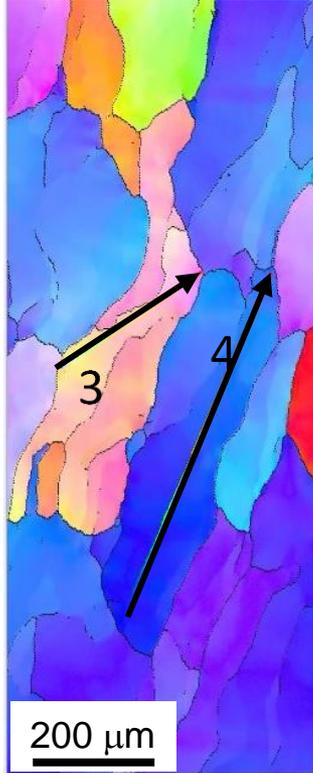
Grain size decreases with increasing preheating temperature
⇒ grain size increases with increasing temperature gradient

Fe-28Al: Misorientations within individual grains

SLM



LMD

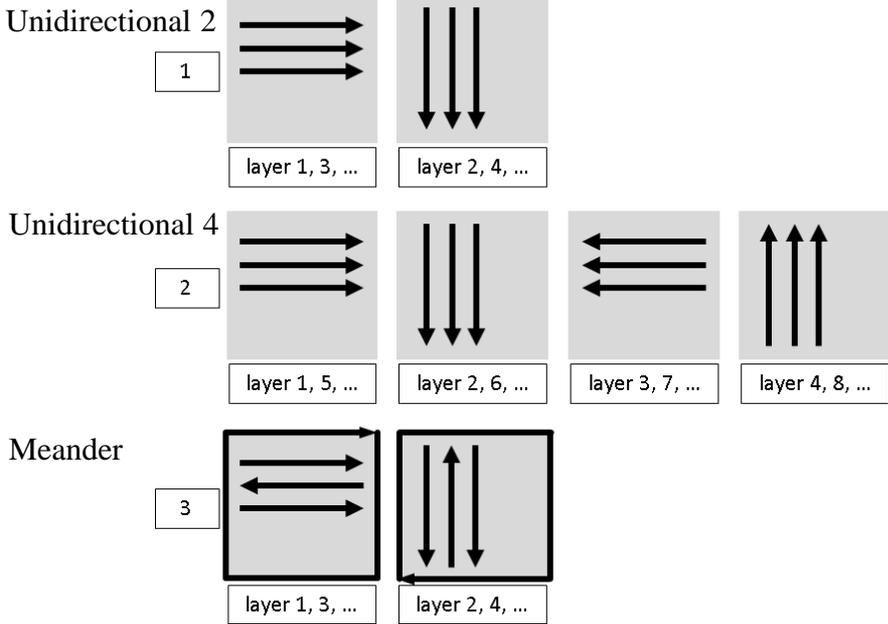


Misorientations within individual grains up to 20°

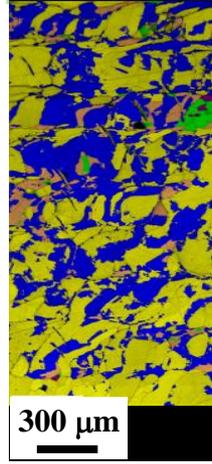
Higher misorientations in SLM compared to LMD (due to higher cooling rate)

Fe-28Al: Effect of scanning strategy on misorientations

SLM; processed at RT

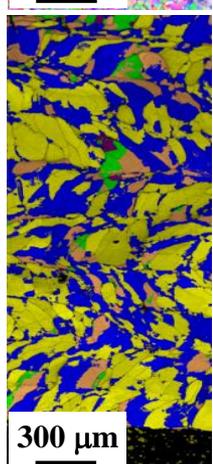


1



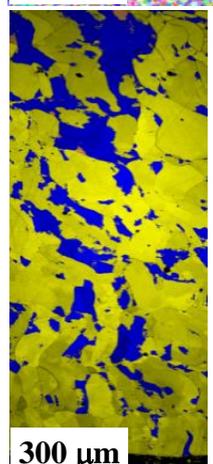
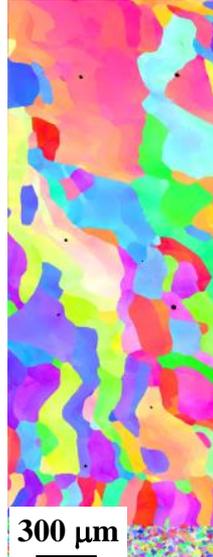
Max.: 15.2°
Av.: 3.2°

2



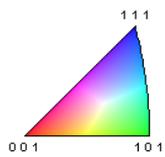
Max.: 16.9°
Av.: 4.0°

3



Max.: 11.9°
Av.: 2.8°

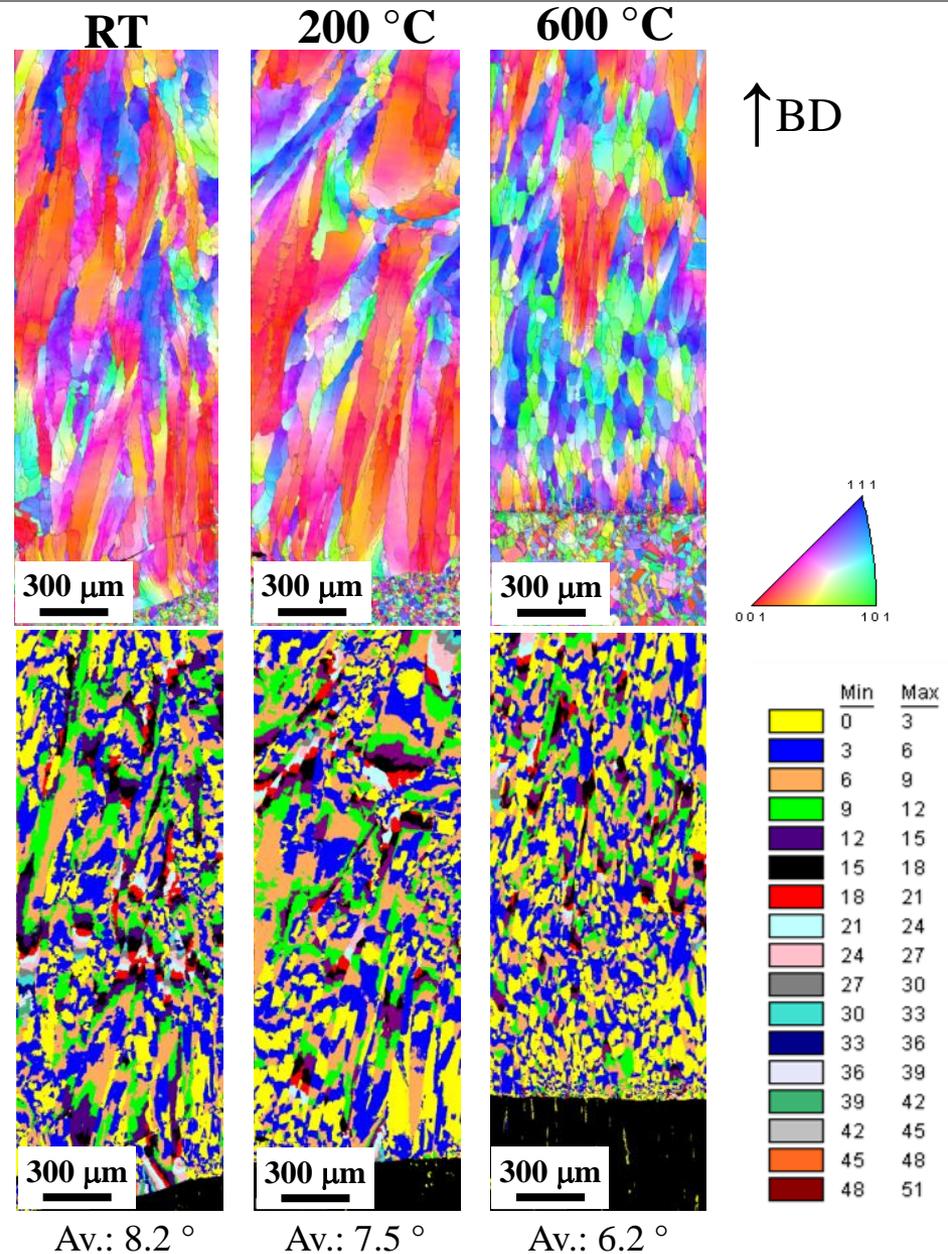
↑BD

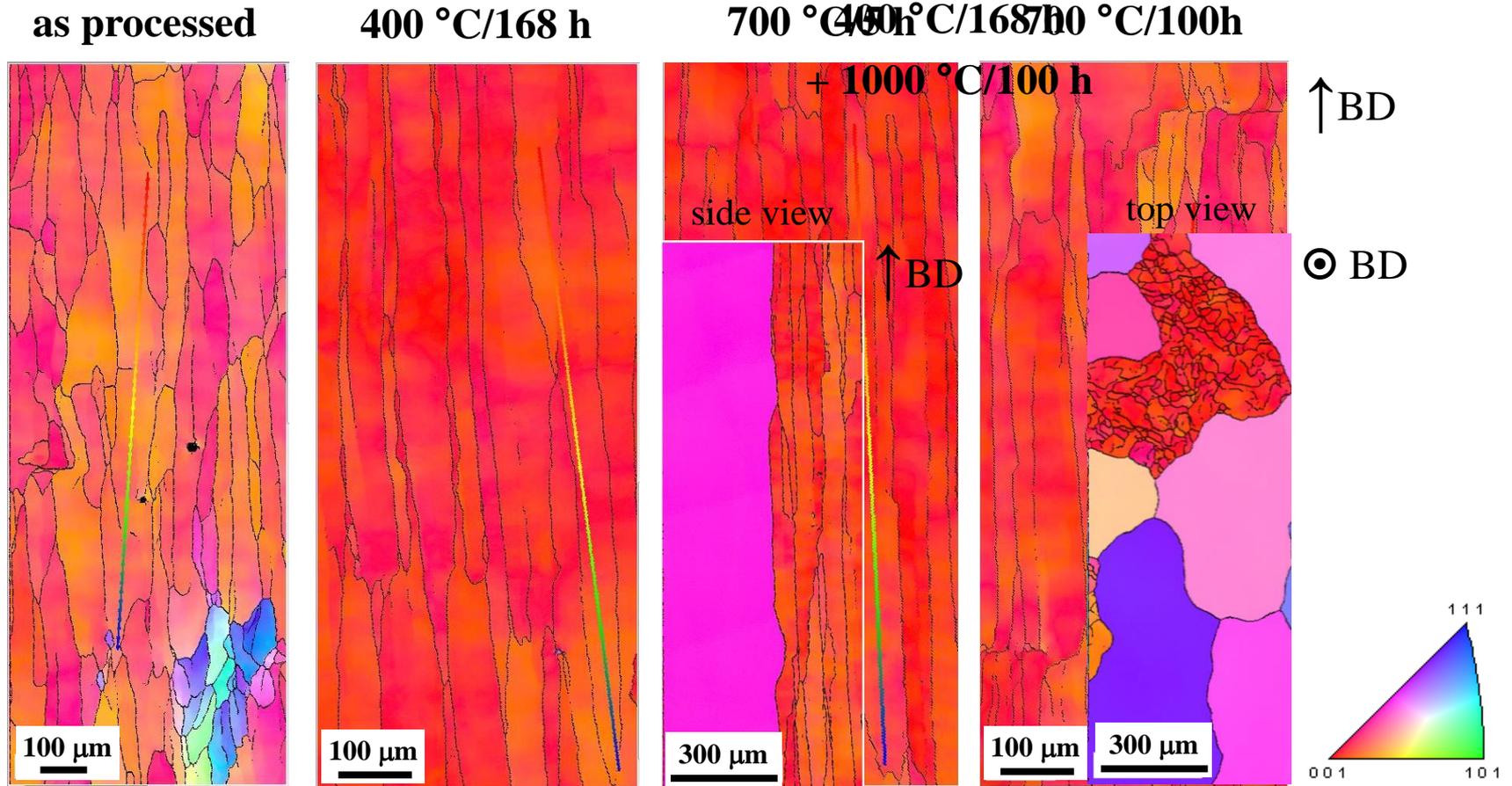


Scan strategy has a marked influence on misorientations



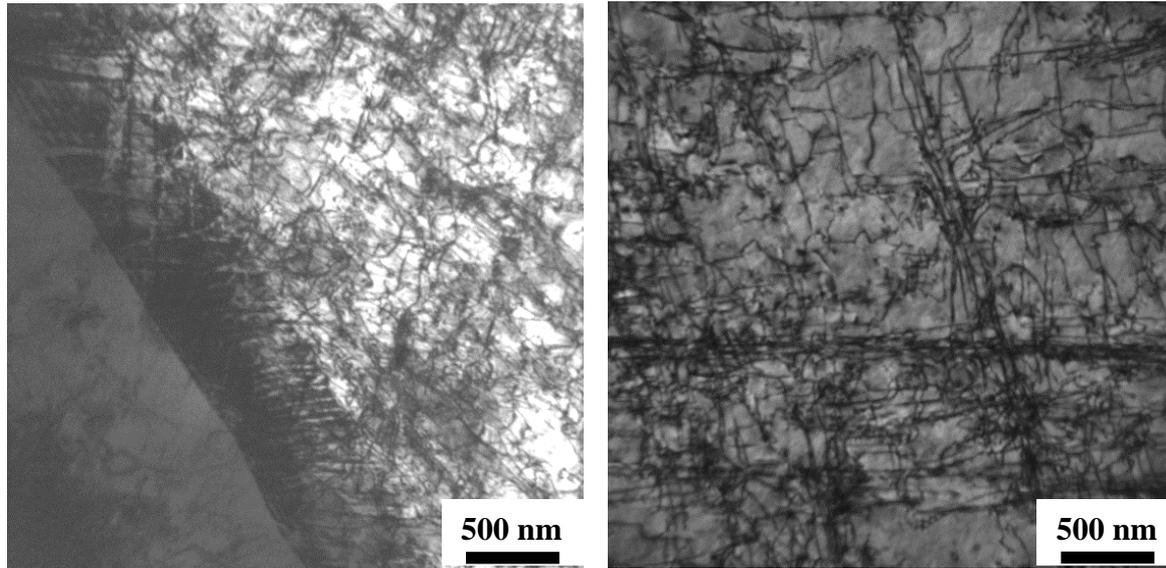
Misorientations decrease with increasing preheating temperature





No recrystallization after various heat treatments

TEM-BF



Sample	Dislocation density
As cast	$3.8 \times 10^{12} \text{ m}^{-2}$
LMD	$2.0 \times 10^{13} \text{ m}^{-2}$
LMD + 700 °C/5 h	$5.8 \times 10^{12} \text{ m}^{-2}$
SLM (KEG)	$2.2 \times 10^{14} \text{ m}^{-2}$
SLM (ILT)	$3.5 \times 10^{14} \text{ m}^{-2}$
SLM + 700 °C/5 h (ILT)	$4.0 \times 10^{13} \text{ m}^{-2}$

Very high dislocation densities

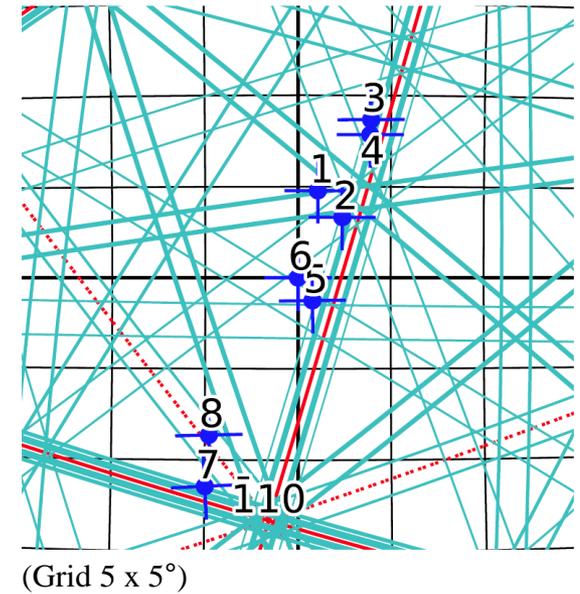
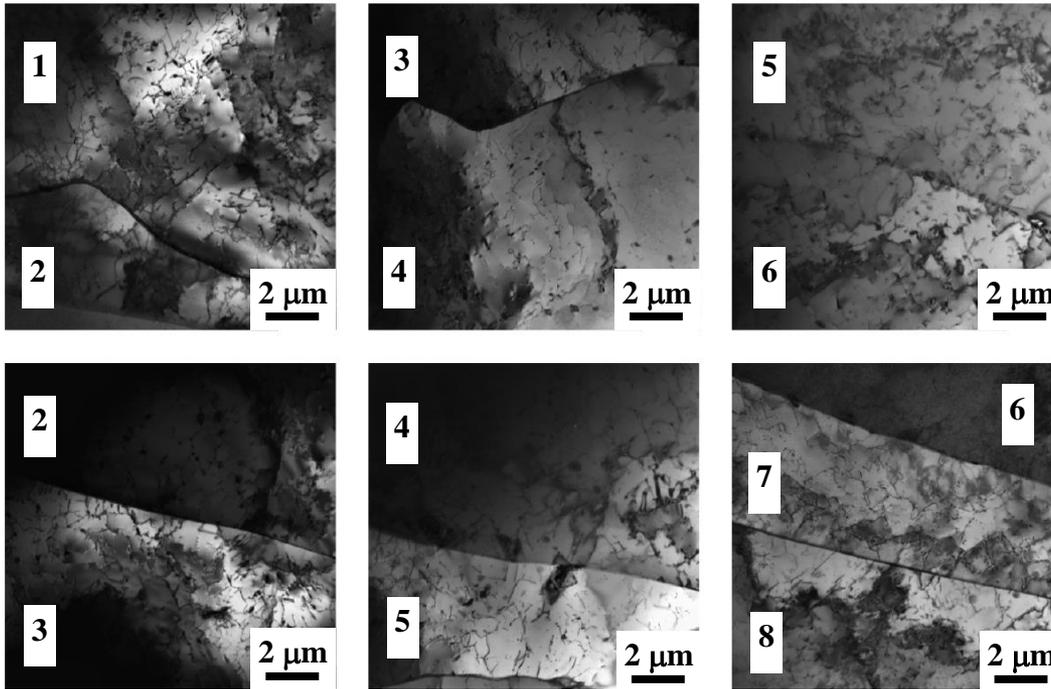
Subgrains are formed through formation of dislocation walls

Dislocations are arranged in a network of parallel screw dislocations

Dislocation densities are about one magnitude higher in SLM samples than in LMD samples

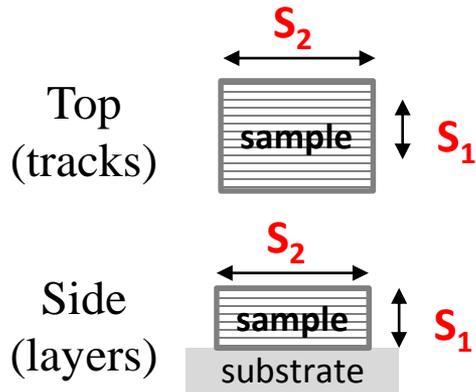
Fe-28Al: TEM analysis of individual subgrains

TEM-BF



Subgrains are located on common zone axes and are inclined by 1–2° against each other.

XRD: local stress measurements



Strategy		Stress S1 [MPa]		Stress S2 [MPa]
LMD 1, top	⊥ tracks	130 ± 60	∥ tracks	36 ± 67
LMD 2, top	⊥ tracks	82 ± 29	∥ tracks	29 ± 70
LMD 3, top	⊥ tracks	109 ± 37	∥ tracks	-10 ± 103
LMD 1, side	⊥ layers	-101 ± 90	∥ layers	84 ± 95
LMD 2, side	⊥ layers	-148 ± 66	∥ layers	-65 ± 37
LMD 3, side	⊥ layers	24 ± 104	∥ layers	16 ± 27
SLM side (20 °C)	⊥ layers	432 ± 172	∥ layers	159 ± 45
SLM side (200 °C)	⊥ layers	378 ± 92	∥ layers	187 ± 27

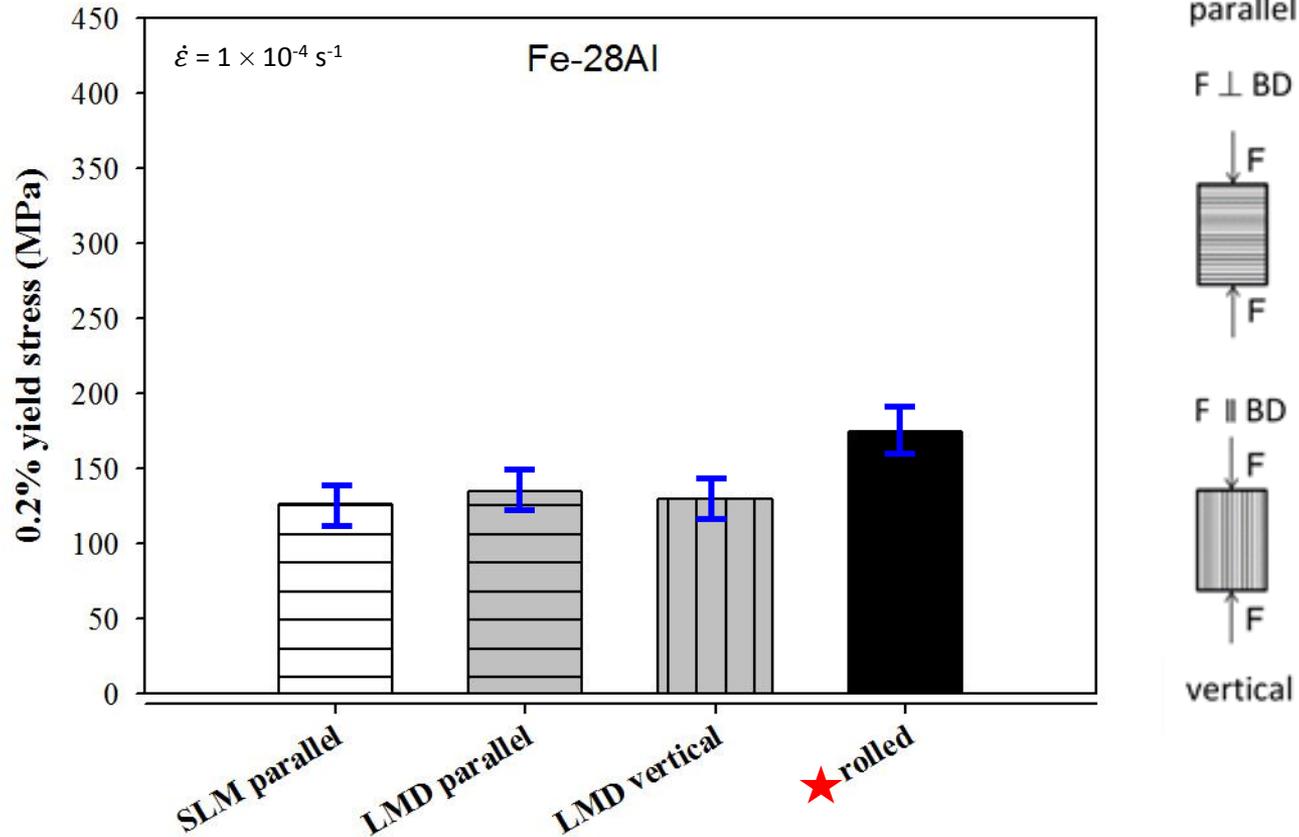
Local stress measurements show mixture of **tensile** and **compressive** stresses.

High **tensile** stresses vertical to individual tracks, low stresses along tracks.

Compressive stresses vertical to individual layers, low stresses along layers.

Stresses are higher in SLM than in LMD samples.

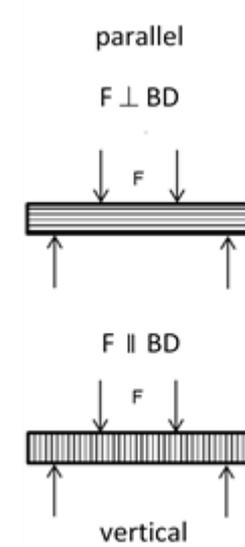
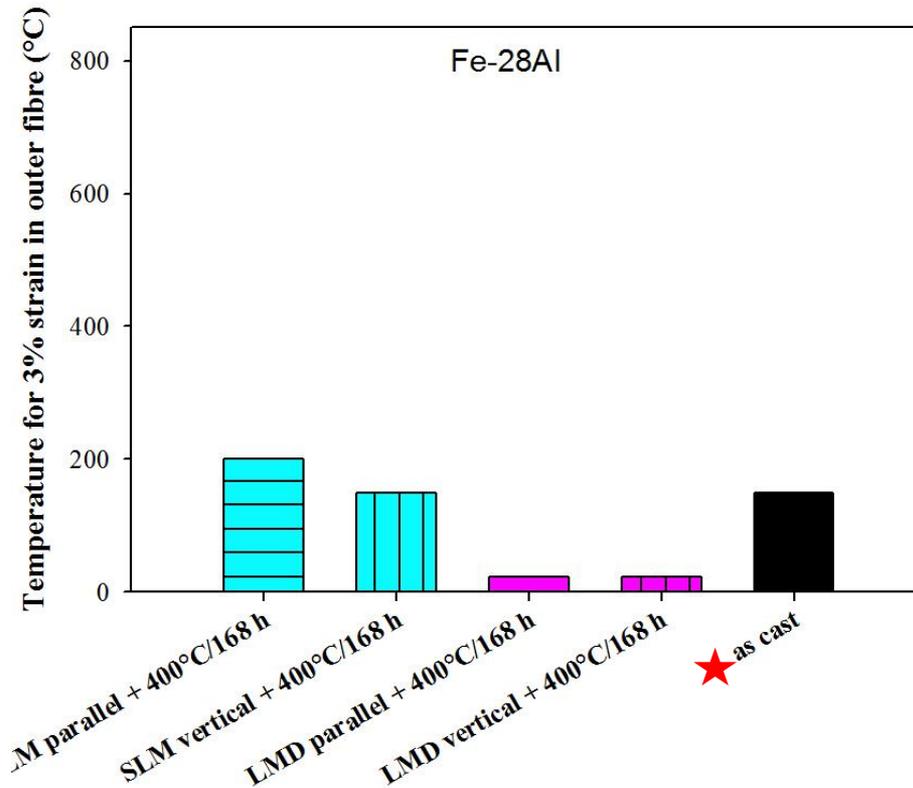
Compressive yield stress of Fe-28Al at 700 °C



No marked difference in compressive yield strength between AM and as-cast samples or whether SLM and LMD samples are tested horizontal (parallel) or vertical to BD.

★ C.G. McKamey et al. ORNL Rep. 10793 (1988) 1-55

Ductility of Fe-28Al determined in 4-point bending



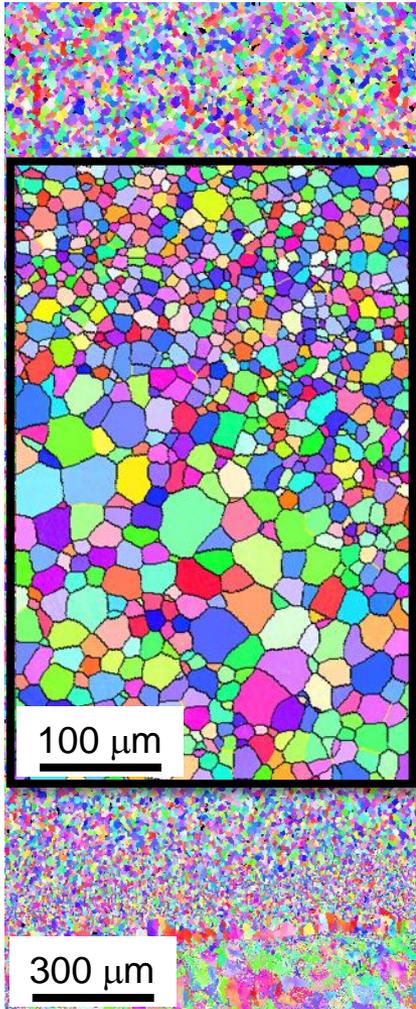
LMD samples show improved ductility compared to SLM and as-cast samples.

The strong crystallographic texture and high dislocation density have no influence on ductility.

Fe–30Al–10Ti: Microstructure

Fe–30Al–10Ti

Increase of $L2_1 \leftrightarrow B2$



↑BD

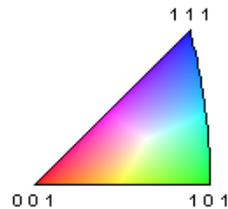
Defect free samples by preheating
at 700 °C (LMD) and 800 °C (SLM)

Equiaxed grains

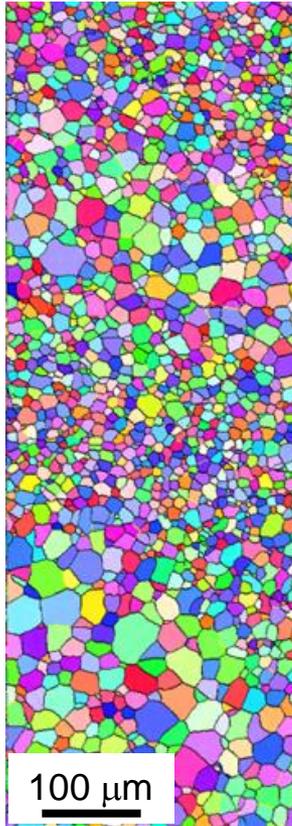
Average grain size < 5 μm (as cast > 100 μm)

No preferred orientation of the grains

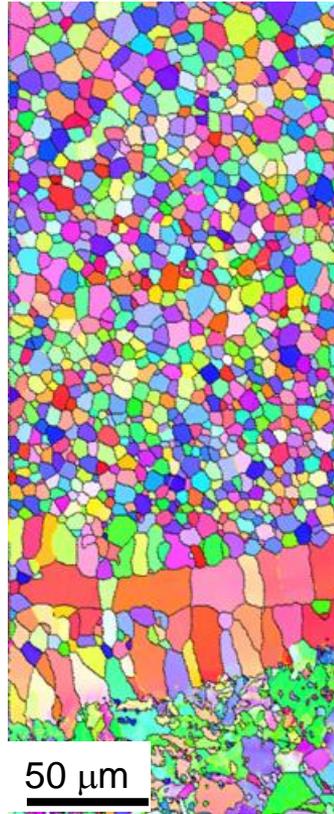
No misorientations within individual grains



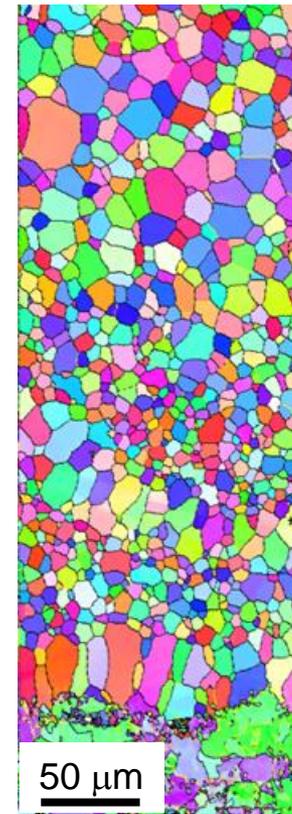
Without preheating



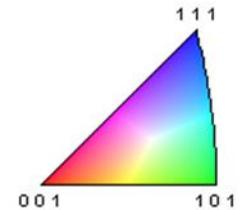
Preheating 700 °C



Preheating 800 °C

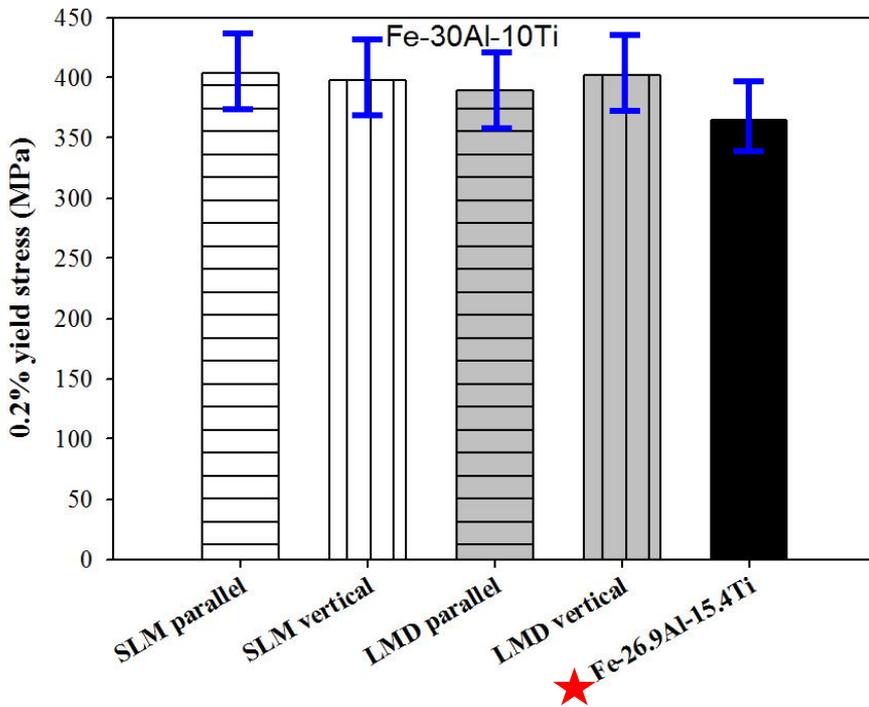


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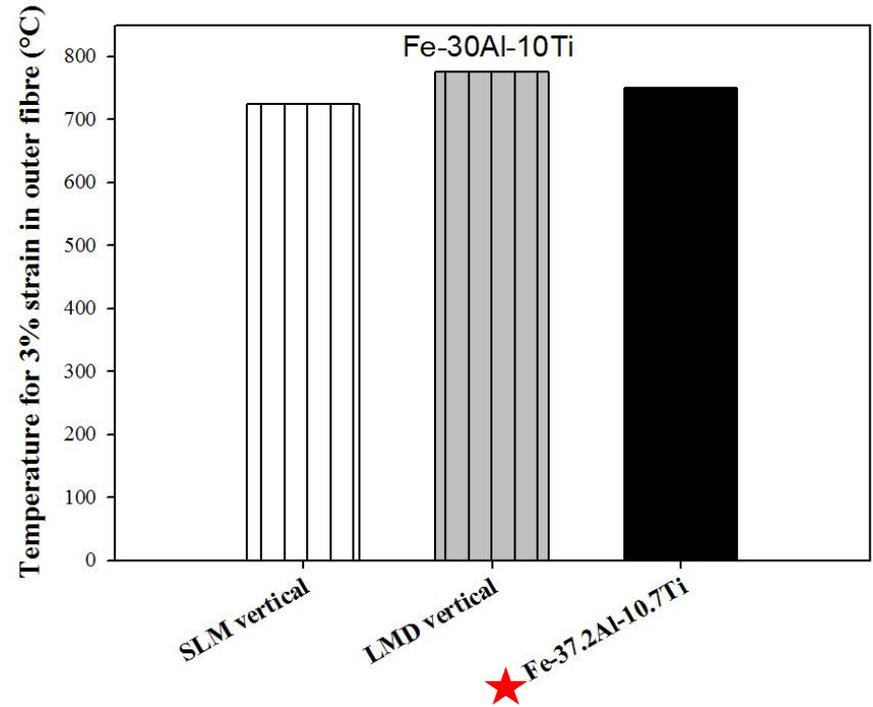


No effect of preheating temperature on grain size

Compressive yield stress of Fe-30Al-10Ti at 700 °C



Ductility of Fe-30Al-10Ti determined in 4-point bending



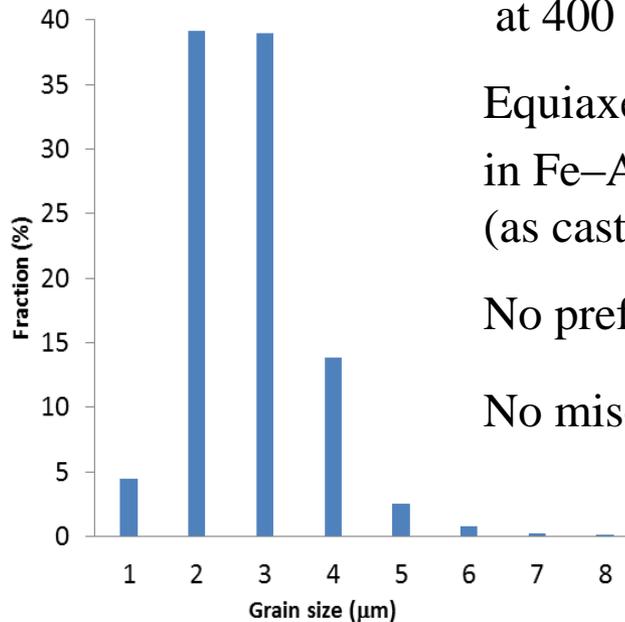
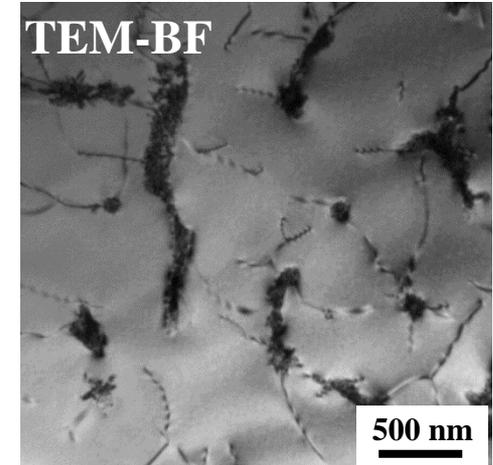
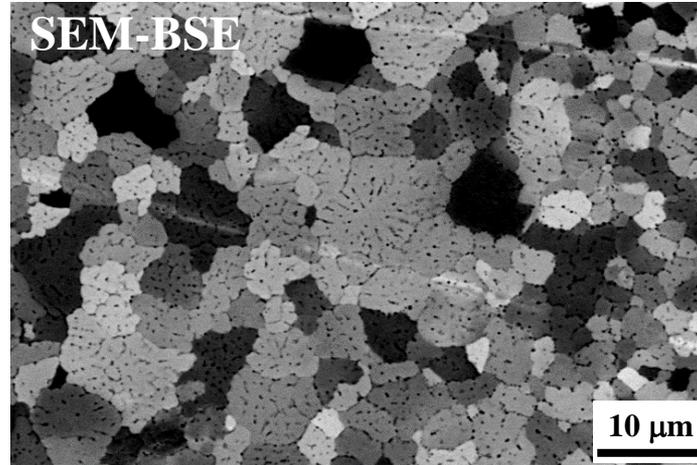
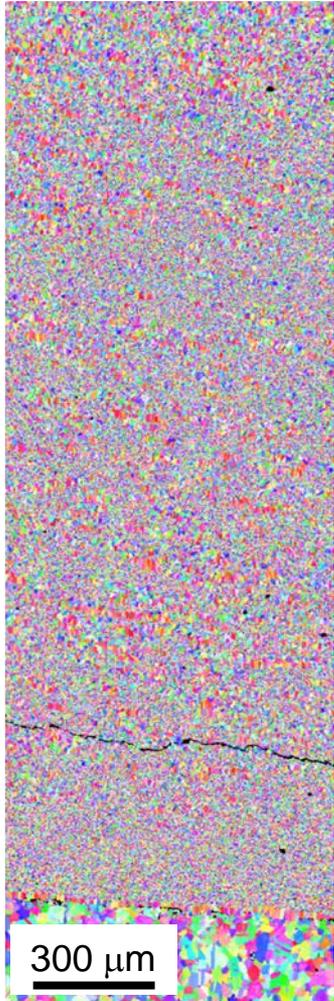
No marked difference in compressive yield strength and ductility between AM and as-cast samples or whether SLM and LMD samples are tested horizontal (parallel) or vertical to BD (yield stress).

★ M. Palm, G. Sauthoff; Intermetallics 12 (2004) 1345

Fe-30Al-5Ti-0.7B: Microstructure

Fe-30Al-5Ti-0.7B

Precipitation of borides



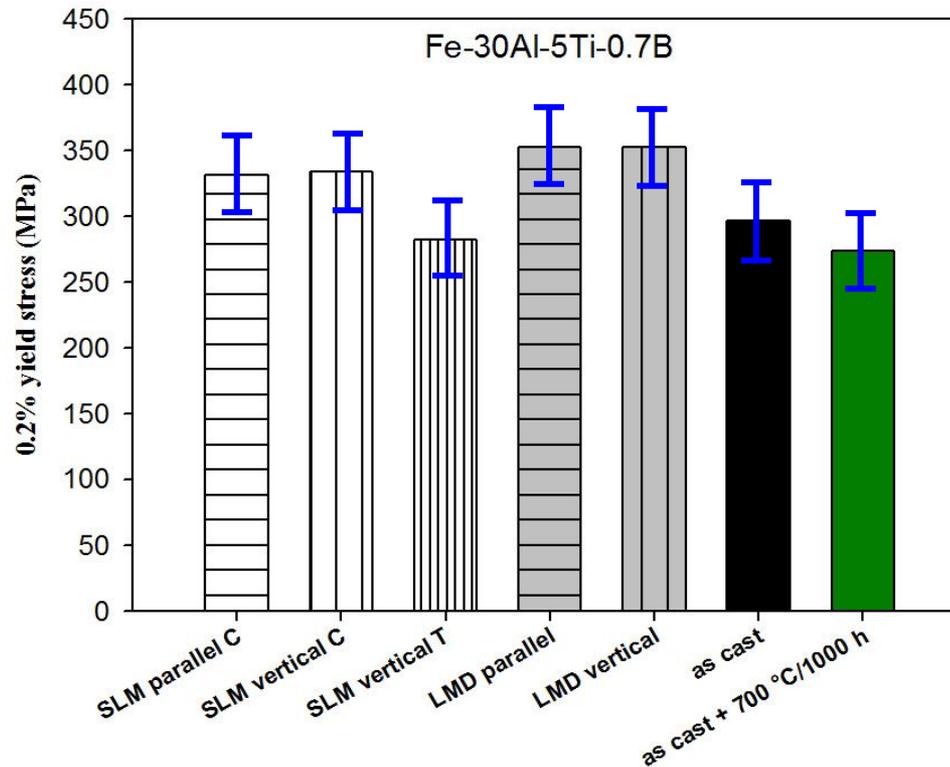
Defect free samples by preheating at 400 °C (LMD) and 600 °C (SLM)

Equiaxed Fe-Al grains (< 5 μm) + TiB₂ (50 nm) in Fe-Al grains and at grain boundaries (as cast: Fe-Al (10 μm) + TiB₂ (100 nm) at GB)

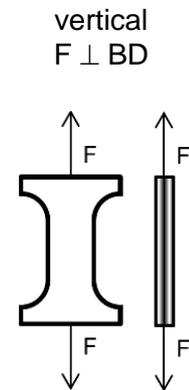
No preferred orientation of the grains

No misorientations within individual grains

Yield stress of Fe-30Al-5Ti-0.7B at 700 °C

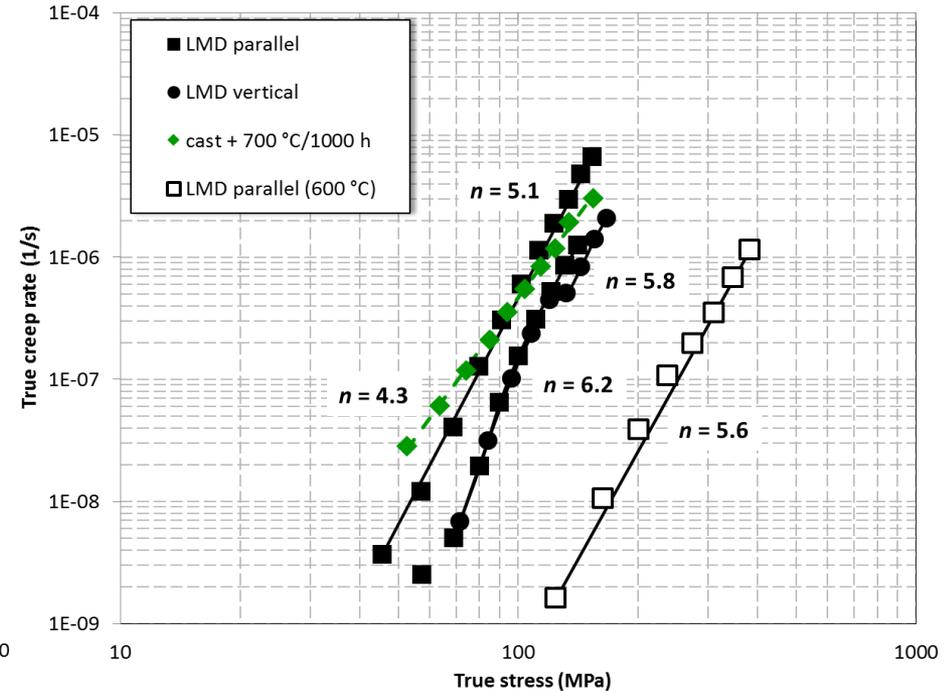
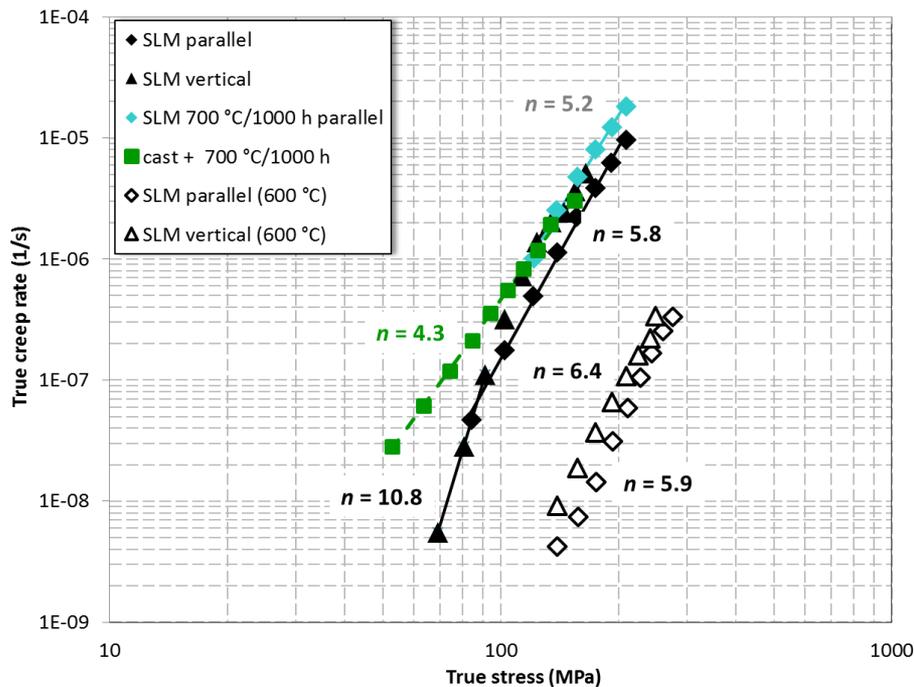


Orientation of the samples for tension (T) testing



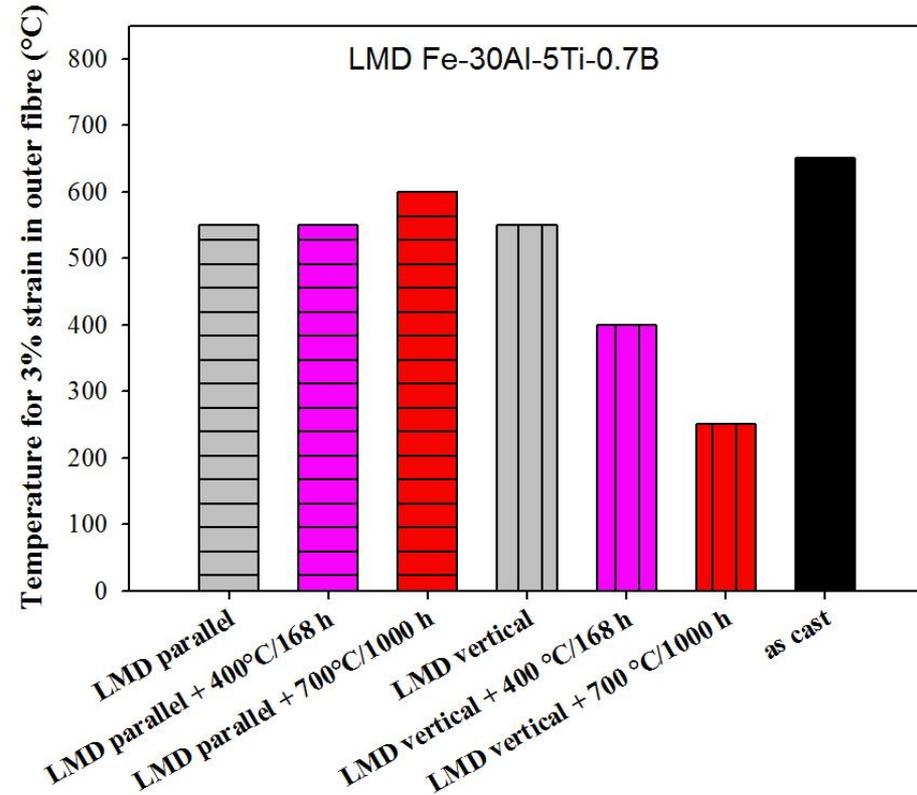
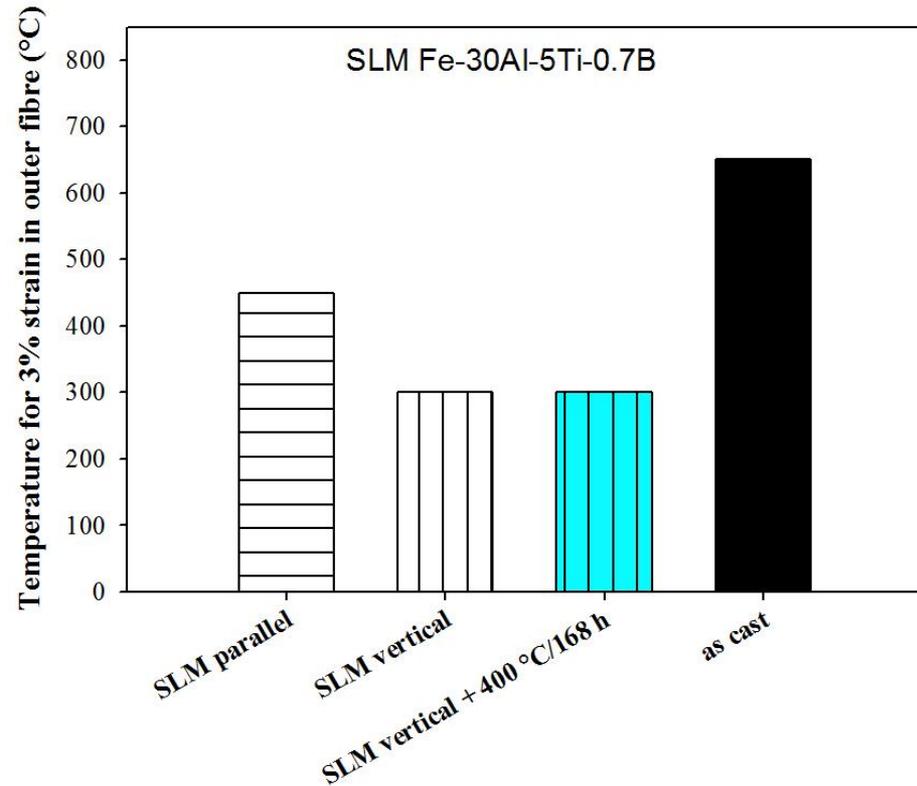
No marked difference in yield strength between AM and as-cast samples or whether SLM and LMD samples are tested horizontal (parallel) or vertical to BD.

Secondary creep of SLM and LMD samples of Fe-30Al-5Ti-0.7B at 600 °C and 700 °C established in compression by stepwise increasing the load



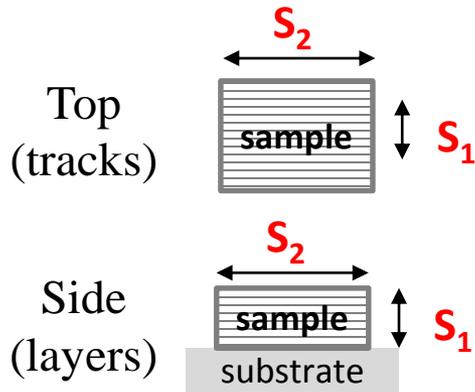
No marked difference in compressive creep strength between AM and as-cast samples or whether SLM and LMD samples are tested horizontal (parallel) or vertical to BD.

Ductility of Fe-30Al-5Ti-0.7B determined in 4-point bending



Marked difference in ductility between SLM and LMD or as-cast samples, whether SLM samples are tested horizontal (parallel) or vertical to BD and if LMD samples are annealed.

XRD: local stress measurements



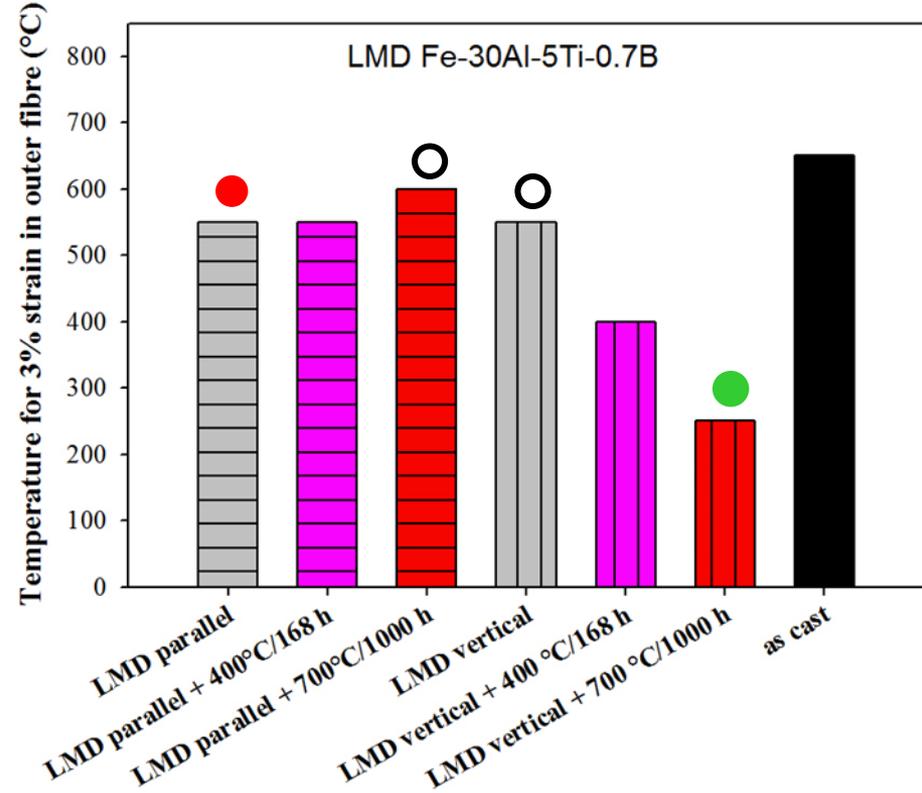
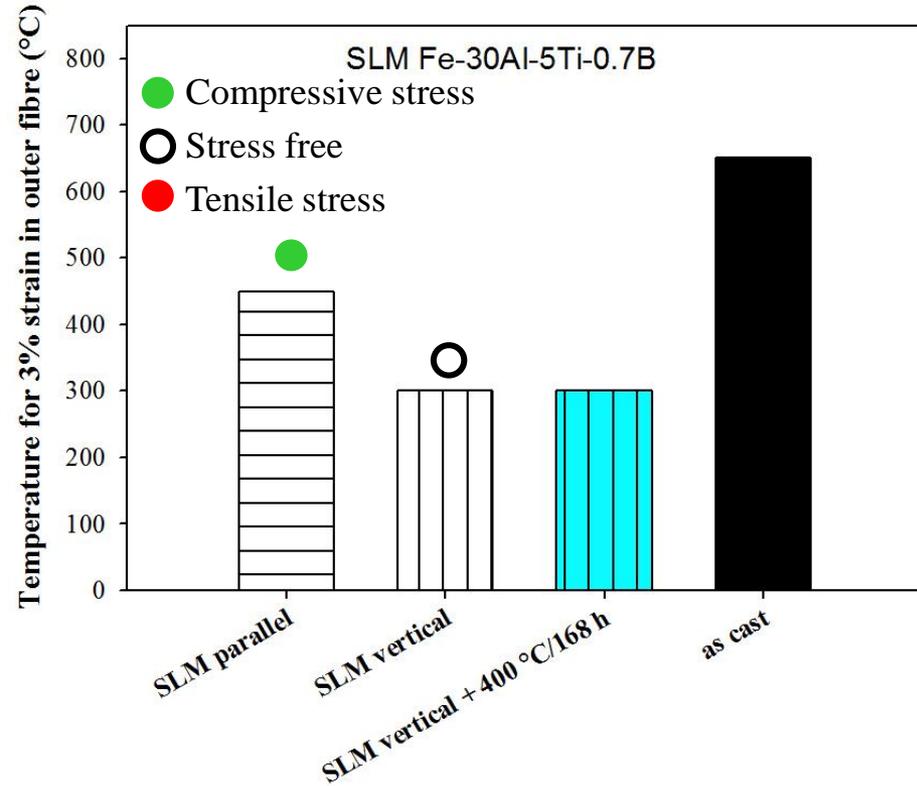
Material/condition		Stress S_1 (MPa)		Stress S_2 (MPa)
LMD top, as processed	⊥ tracks	114 ± 12	∥ tracks	-32 ± 11
LMD side, as processed	⊥ layers	-109 ± 36	∥ layers	5 ± 8
LMD top (700 °C/1000 h)	⊥ tracks	-22 ± 2	∥ tracks	-36 ± 2
LMD side (700 °C/1000 h)	⊥ layers	-73 ± 5	∥ layers	-69 ± 6
SLM top, as processed	⊥ tracks	-87 ± 4	∥ tracks	38 ± 4
SLM side, as processed	⊥ layers	79 ± 9	∥ layers	8 ± 10
SLM top (700 °C/1000 h)	⊥ tracks	-4 ± 9	∥ tracks	10 ± 6
SLM side (700 °C/1000 h)	⊥ layers	-46 ± 3	∥ layers	-35 ± 3

Local stress measurements show mixture of moderate **tensile** and **compressive** stresses.

Tensile or **compressive** (SLM) stresses vertical to individual tracks, low stresses along tracks.

Compressive stresses vertical to individual layers, low stresses along layers.

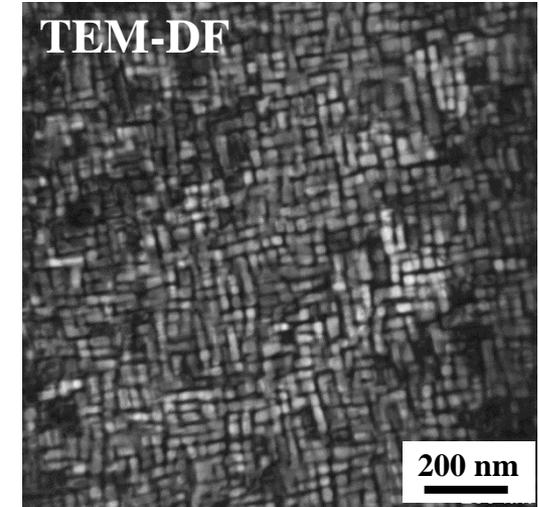
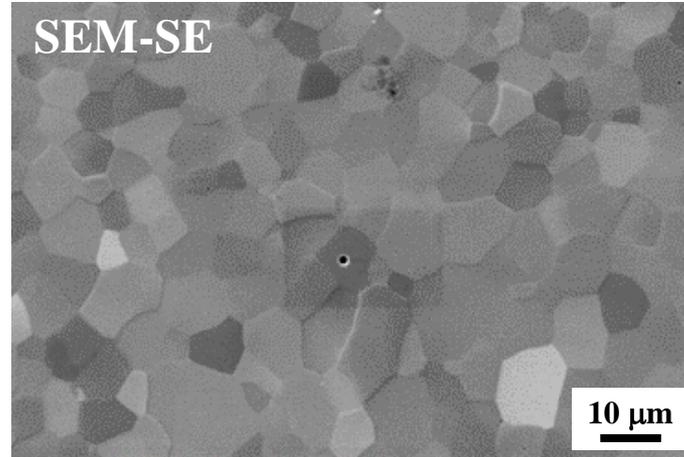
Ductility of Fe-30Al-5Ti-0.7B determined in 4-point bending



No direct relation between internal stresses and ductility of the samples.

Fe-22Al-5Ti : Microstructure

Fe-22Al-5Ti
Coherent A2+ L2₁

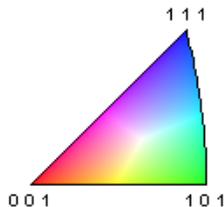


Defect free samples by preheating at $>800\text{ }^{\circ}\text{C}$ (LMD) and $800\text{ }^{\circ}\text{C}$ (SLM)

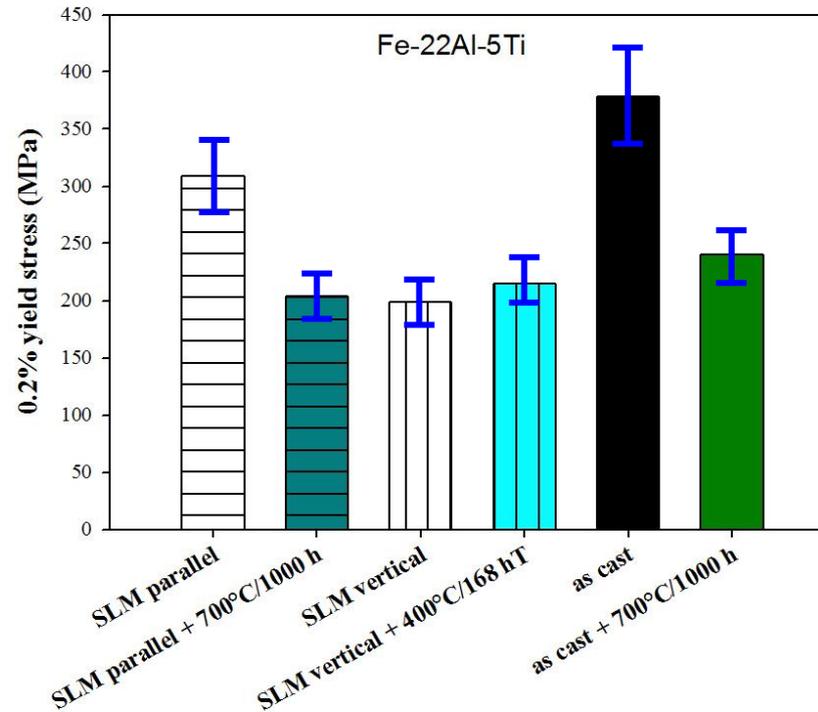
Equiaxed grains ($10\text{ }\mu\text{m}$) with coherent A2 + L2₁ microstructure (30 nm)
(comparable to as-cast microstructure)

No preferred orientation of the grains

No misorientations within individual grains

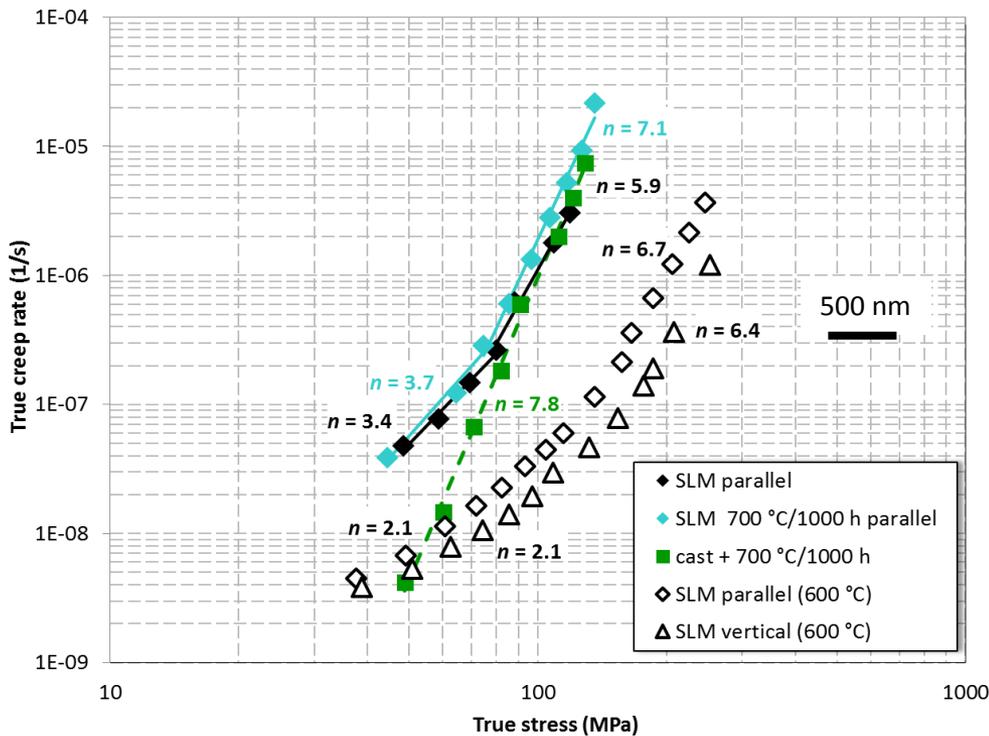


Compressive yield stress of Fe-22Al-5Ti at 700 °C

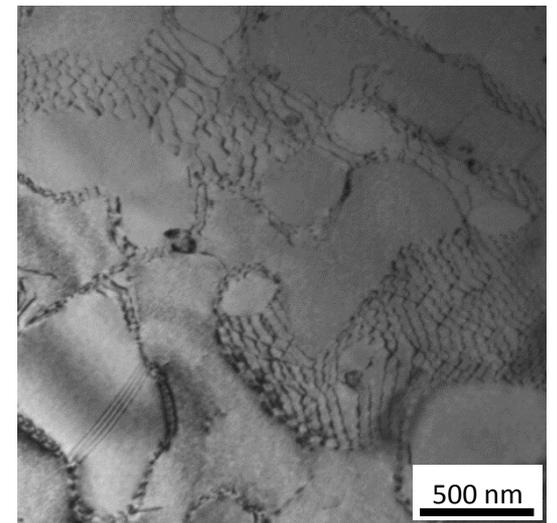
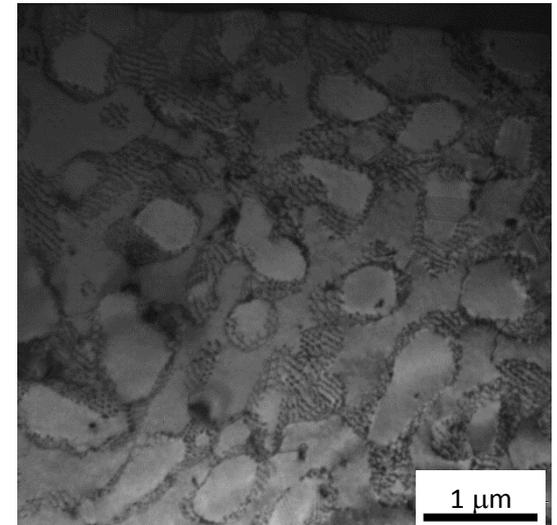


Marked difference whether SLM samples are tested horizontal or vertical to BD.
Reduction of yield strength after annealing at 700 °C due to coarsening of coherent microstructure.

Secondary creep of SLM samples of Fe-22Al-5Ti at 600 °C and 700 °C established in compression by stepwise increasing the load



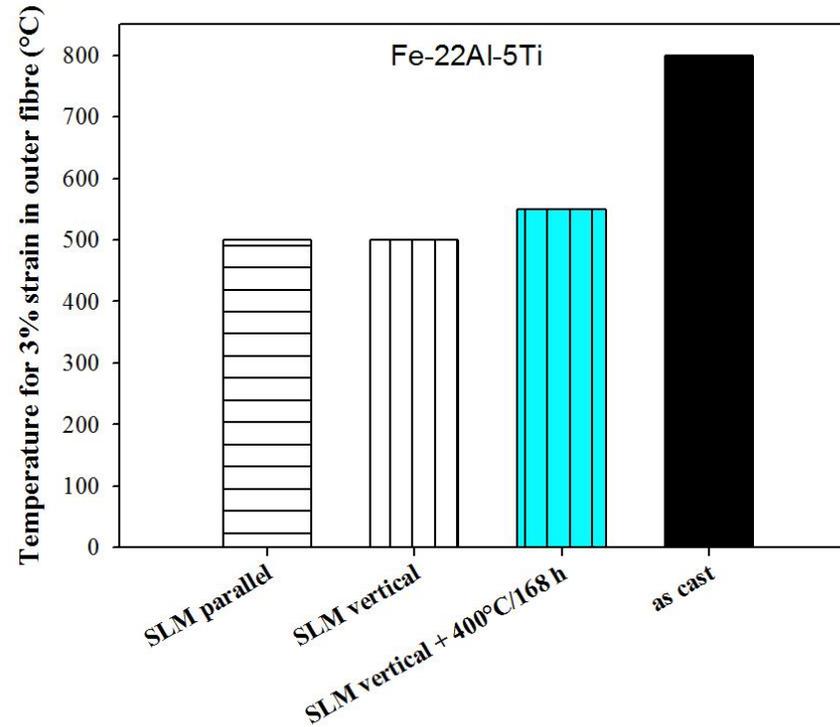
700 °C/ 100 h (TEM-BF)



Formation of dislocation networks

No marked difference in compressive creep strength between SLM and as-cast samples or whether SLM samples are tested horizontal (parallel) or vertical to BD.

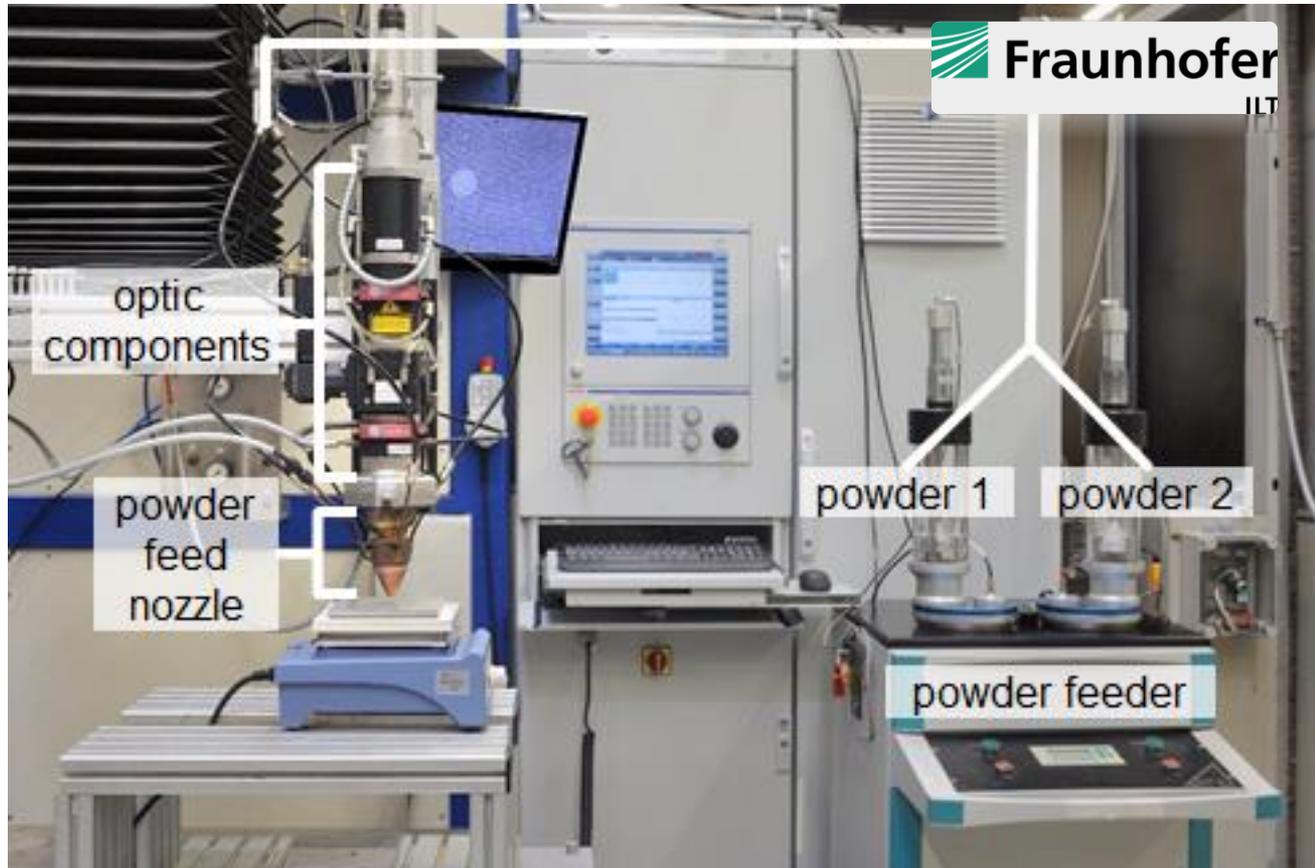
Ductility of Fe-22Al-5Ti determined in 4-point bending



SLM samples show improved ductility compared to as-cast samples.



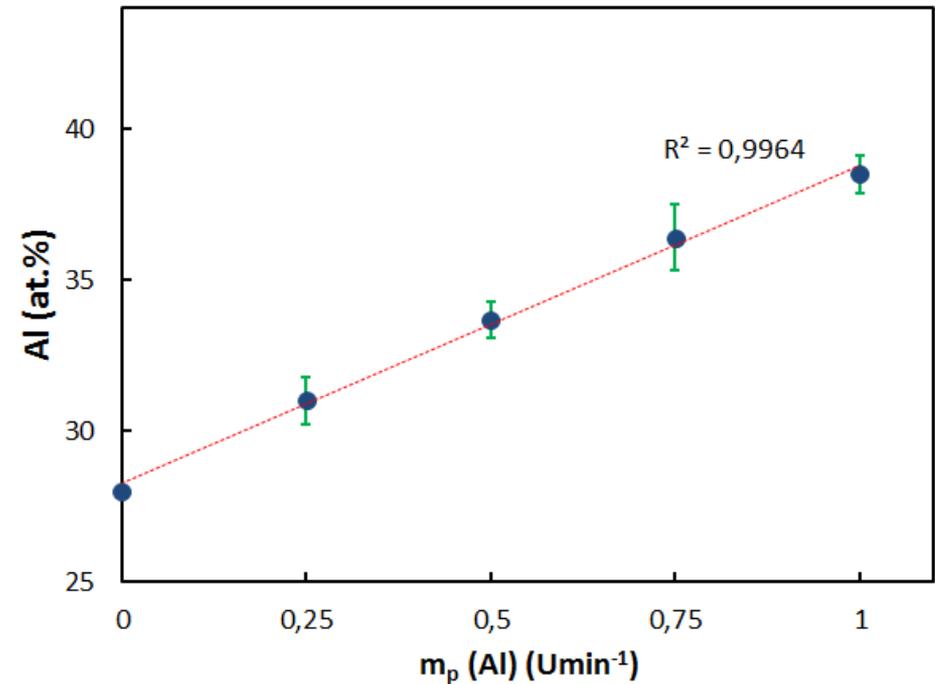
Chemically graded Fe–Al samples produced by LMD



- YAG-Laser: 2000 W
- Fe-28Al: powder particle size 45 – 90 μm
- Chemical composition varied by the rotation speed m_p (Umin^{-1}) of the feeder

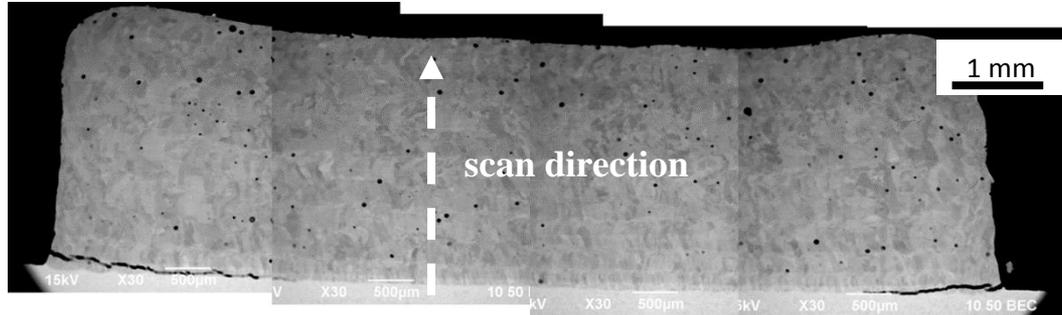
Production of binary Fe–Al samples by varying the speed (m_p) of the Al feeder

m_p Al (Umin^{-1})	Al (at%)
0.25	31.0 ± 0.8
0.50	33.7 ± 0.6
0.75	36.4 ± 1.1
1.00	38.5 ± 0.6

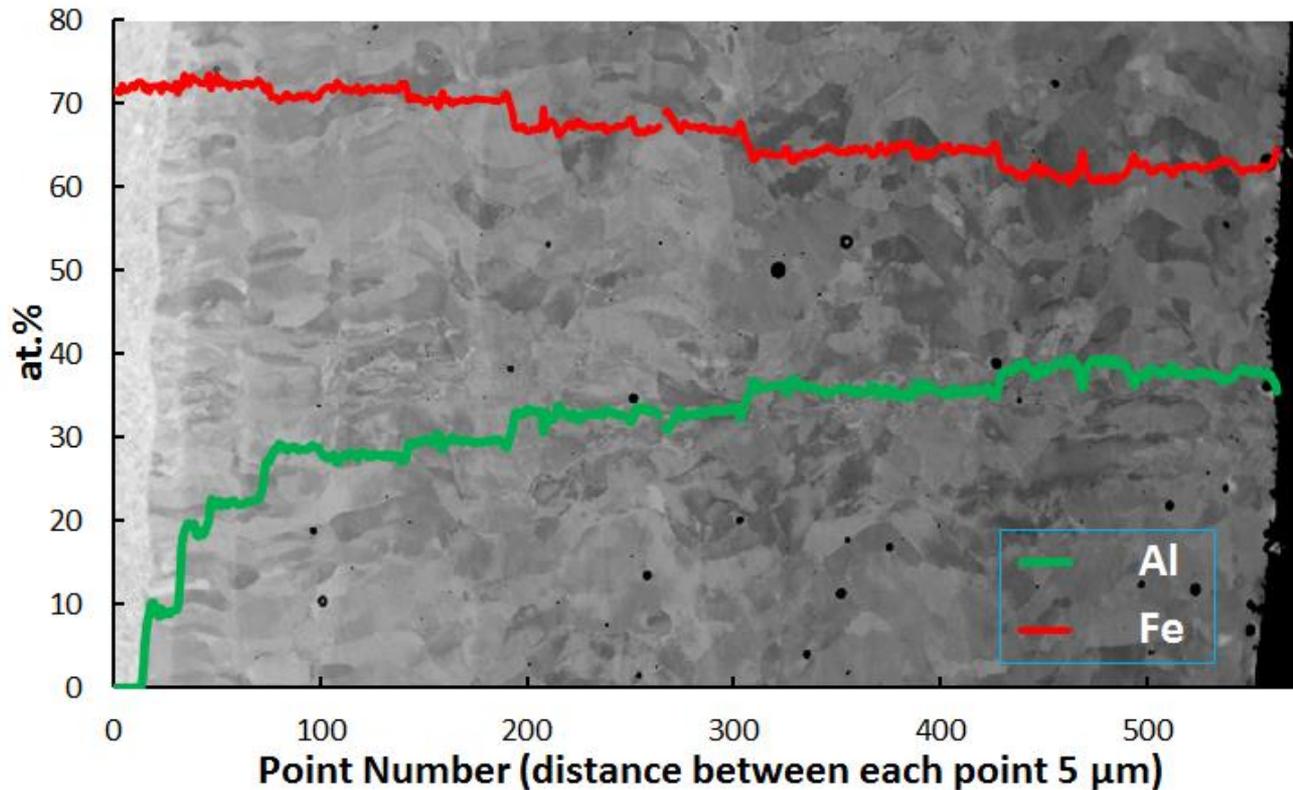


Linear dependence between rotation speed of the feeder and obtained composition.

⇒ Production of LMD samples with defined composition gradients possible

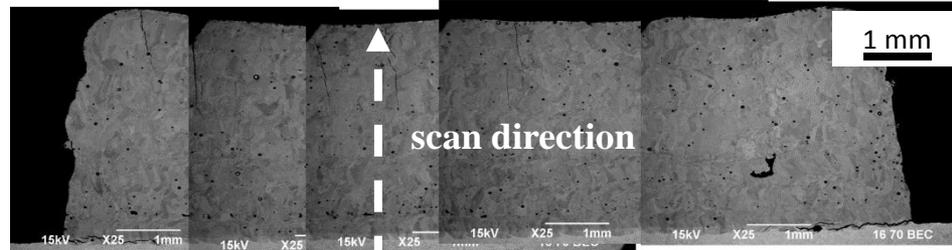


SEM-EDS

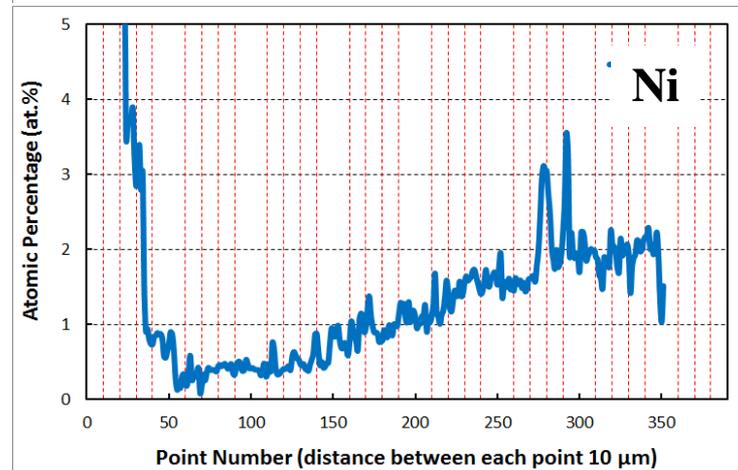
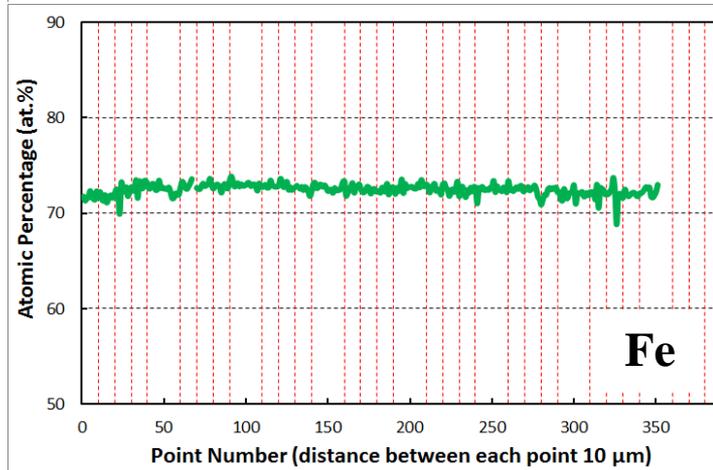
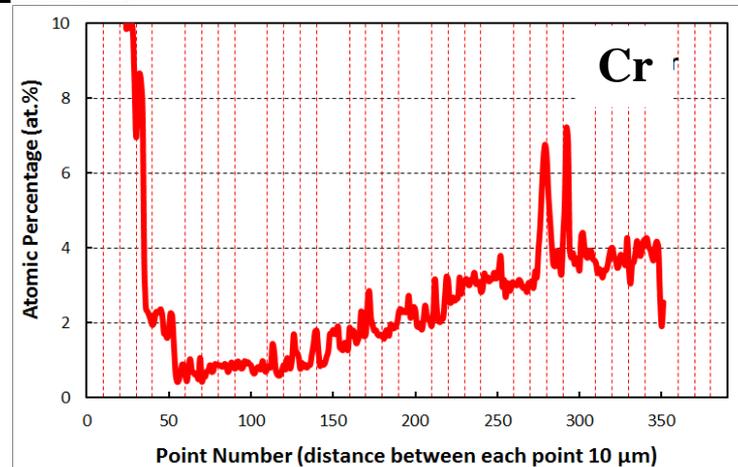
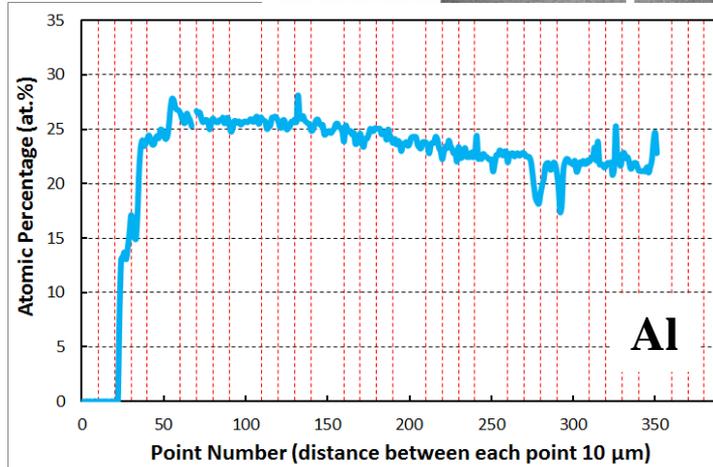


Production of chemically graded Fe–Al samples by LMD is possible.

Chemically graded Fe–Al/ steel 316L sample



EPMA (WDS)



Production of chemically graded Fe–Al/steel samples by LMD is possible.



- Intermetallic Fe–Al alloys can be processed by SLM and LMD
- Fe–Al alloy concepts developed for casting can be transferred to AM
- Yield strength and creep strength are comparable to that of as cast alloys
- Partly improvement of ductility (through internal stresses (?) not through reduction of grain size)
- Chemically graded Fe–Al samples with defined concentration profiles can be manufactured by LMD

MPIE

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