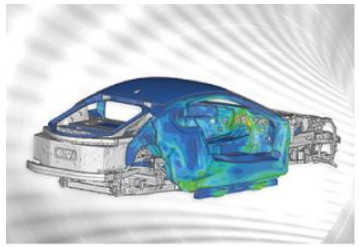

AUTOMOTIVE


INFOCOM


MOBILITY, ENERGY & ENVIRONMENT


AERONAUTICS


SPACE


DEFENCE & SECURITY

IABG - Final presentation

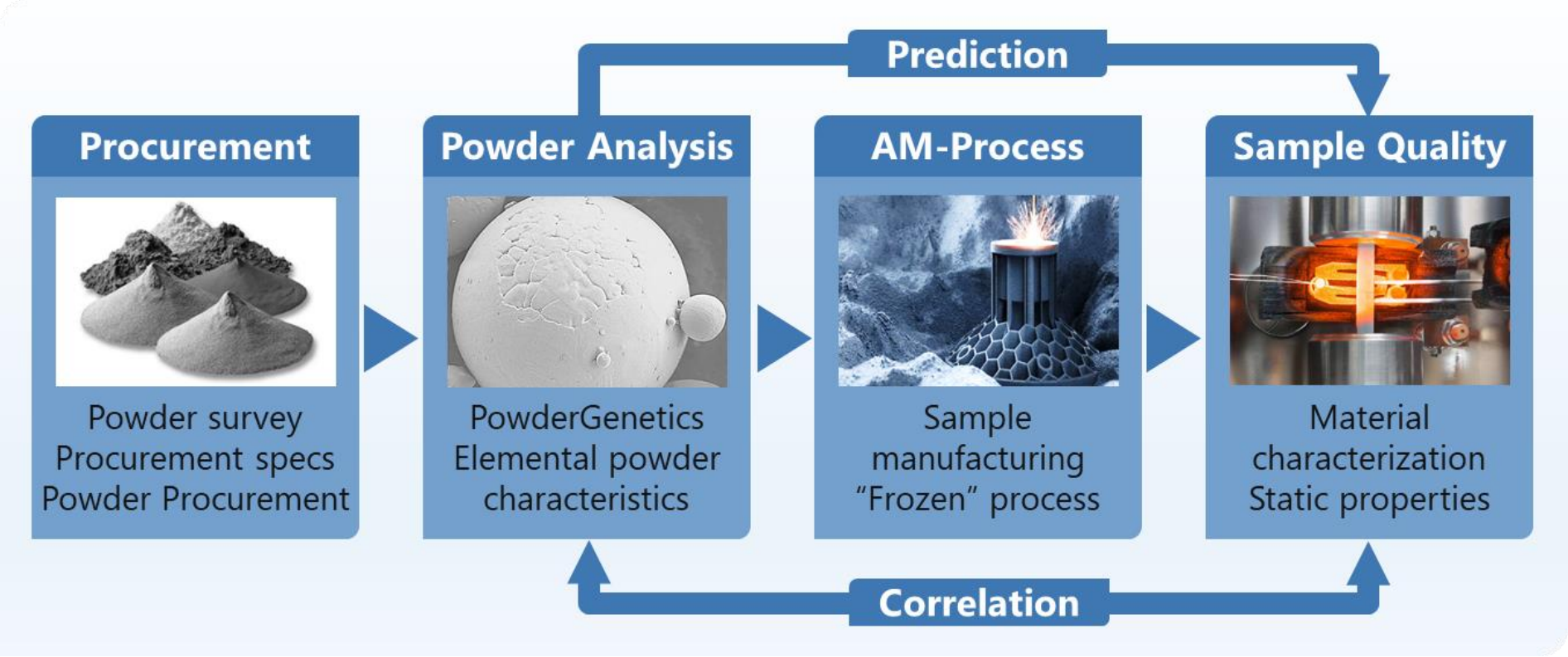
„Assessments to Prepare and De-Risk Technology Developments – Powder Characterisation“

Industrieanlagen-Betriebsgesellschaft mbH
Einsteinstraße 20
D-85521 Ottobrunn

Scope of work

- Evaluation and correlation of powder characteristics with the processability of the powder within the additive manufacturing process and with the respective part quality

- Establish a correlation between powder properties and test specimen quality



WP 2000 – Procurement of Sample Powder Batches

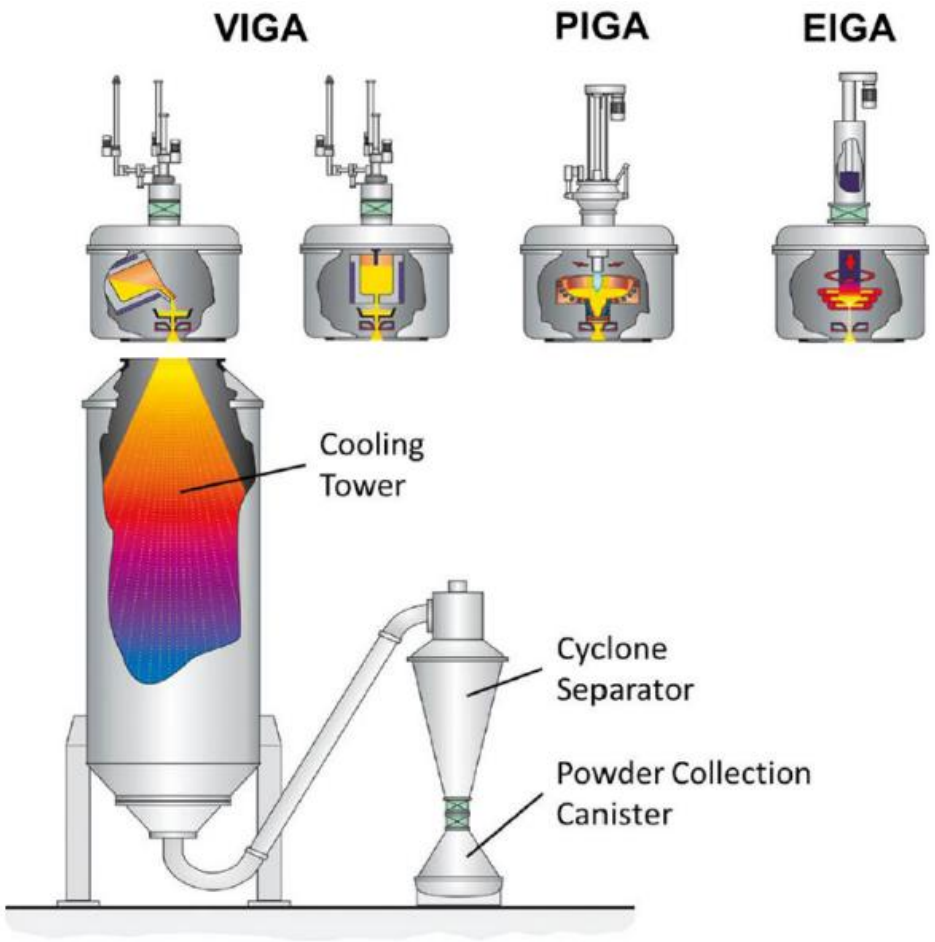
- **IN718 powder landscape for additive manufacturing**

- Three major powder production methods:

  - VIGA
    Vacuum Induction Melting combined with Inert Gas Atomization

  - PIGA
    Plasma Melting Induction Guiding Gas Atomization

  - EIGA
    Electrode Induction Melting Gas Atomization



WP 2000 – Procurement of Sample Powder Batches

- **IN718 powder landscape for additive manufacturing**

- Worldwide survey of powder suppliers
  - Europe: 29
  - North America: 10
  - Asia: 11
  - No suppliers found in Africa, Australia, Central and South America
  - 50 potential companies identified

- Supplier trade-off analysis was performed with the following criteria:
  - powder quality: max. 30 points
  - price: max. 25 points
  - responseness: max. 20 points
  - delivery time: max. 20 points

WP 2000 – Procurement of Sample Powder Batches

- **IN718 powder landscape for additive manufacturing**

- Result of the supplier trade-off analysis:

Ranking	PIGA Manufacturer	Powder Quality	Price	Respon-seness	Delivery Time	Total Result
	Max. Points	35	25	20	20	**100**
1.	TEKNA (CA)	27	7	10	20	**64**
2.	AP & C (CA)	25	19	10	5	**59**

Ranking	VIGA Manufacturer	Powder Quality	Price	Respon-seness	Delivery Time	Total Result
	Max. Points	35	25	20	20	**100**
1.	Praxair (US)	22	25	20	20	**87**
2.	VDM (DE)	27	22	20	15	**84**
3.	Hoeganaes (SE)	16	24	15	20	**75**
4.	CNPC-POWDER (CA)	13	21	10	15	**59**
5.	Carpenter Additive (GB)	17	9	10	20	**56**
6.	Heraeus (DE)	17	17	15	0	**49**
7.	Sandvik (GB)	13	22	10	0	**45**
8.	Oerlikon metco (DE)	22	18	0	0	**40**
9.	TLS-Technik (DE)	10	13	10	5	**38**
10.	Eramet (FR)	17	0	5	0	**22**
11.	NMD (DE)	13	0	0	0	**13**
12.	EOS (FI)	9	0	0	0	**9**
13.	Voestalpine (AT)	8	0	0	0	**8**

- Tekna, AP&C, Praxair and VDM were chosen as supplier

WP 2000 – Procurement of Sample Powder Batches

Powder Procurement Specification

- 1. Production Process
  - VIGA or PIGA
- 2. Material Composition
  - Ni-Alloy IN718 / 2.4668

Element	Minimum in wt-%	Maximum in wt-%
Ni	50.0	55.0
Cr	17.0	21.0
Fe	Balance	Balance
Nb	4.70	5.50
Mo	2.80	3.30
Ti	0.60	1.20
Al	0.30	0.70
Cu	-	0.30
C	0.02	0.08
Si	-	0.35
Mn	-	0.35
B	-	0.006
Co	-	1.00
P	-	0.015
S	-	0.015
All other Elements each	-	0.05
Higher density particles*	-	0.005
Hollow spaces inside the particles**	-	0.07

WP 2000 – Procurement of Sample Powder Batches

Powder Procurement Specification

3. Particle Size Distribution

D-Values of the cumulative volume	Minimum in µm	Maximum in µm
D10	12	24
D50	24	36
D90	36	54

- Determined by: Light diffraction, Light scattering or Image analysis with more than 100000 particles analysed
- Determination by sieving is not accepted

4. Morphology

- Sufficient spherical, sufficient few agglomerates and satellites for the SLM process
- Verified by scanning electron microscope (SEM) images of the powder.
- Optional: sphericity has to be higher than 0.85 (mean value) described by image analysis

WP 2000 – Procurement of Sample Powder Batches

- **Powder Procurement Specification**

- 5. Delivery
  - To Fraunhofer-IGCV in Augsburg
  - within suitable sealed transport container
  - breakage of the seal or entrance of moisture will lead to rejection

- 6. Certificate of Conformity
  - Contains the proof of points:
    - 1. production process
    - 2. material
    - 3. particle size distribution
    - 4. morphology

- 7. Other agreements

WP 2000 – Procurement of Sample Powder Batches

IBL-M sample no.	Powder Manufacturer	Batch number with quantity	Comment
19-210-01	Praxair (VIGA)	Lot# 133 8 containers each 10 kg	Tropagel Desiccant inside; Sealed and numbered by identification number
19-210-02	Tekna (PIGA)	Lot: 39596-MG19019 16 containers each 5 kg	Tropagel Desiccant inside; Sealed and numbered by identification number
19-210-03 Batch 1	AP&C (PIGA)	Lot: 201-J0073 13 containers each 5 kg	No sealing No identification number
19-210-03 Batch 2	AP&C (PIGA)	Lot: 201-J0092 3 containers each 5 kg	No sealing No identification number
19-210-04 Batch 1	VDM (VIGA)	Charge # P10218 2 containers: 19.90 kg and 20.25 kg	No sealing Identification number
19-210-04 Batch 2	VDM (VIGA)	Charge # P10220 2 containers: 19.85 kg and 19.00 kg	No sealing Identification number

- Powder was stored at IGCV in a safety cabinet

WP 3000 – Characterization of Powder Batches

Characterization of powder batches by PowderGenetics

Powder manufacturers provided material certificates

Tested Powder Properties	19-210-01 Praxair (VIGA)	19-210-02 Tekna (PIGA)	19-210-03 AP&C (PIGA)	19-210-04 VDM (VIGA)
Chemical composition	✓	✓	✓	✓
Particle Size Distribution	✓	✓	✓	✓
Morphology	✕	✓	✓	✕
Hollow space in particles	✕	✕	✕	B1: 0.03 % B2: 0.08 %
Apparent density	4.21 g / cm³	4.59 g / cm³	B1: 4.57 g / cm³ B2: 4.58 g / cm³	✕
Tap density	5.01 g / cm³	5.2 g / cm³	✕	✕
Flow rate	14 s	11 s	B1: 2.64 s B2: 2.2 s	✕
Addition feature	Visual inspection	-	LOM picture	SEM pictures

WP 3000 – Characterization of Powder Batches

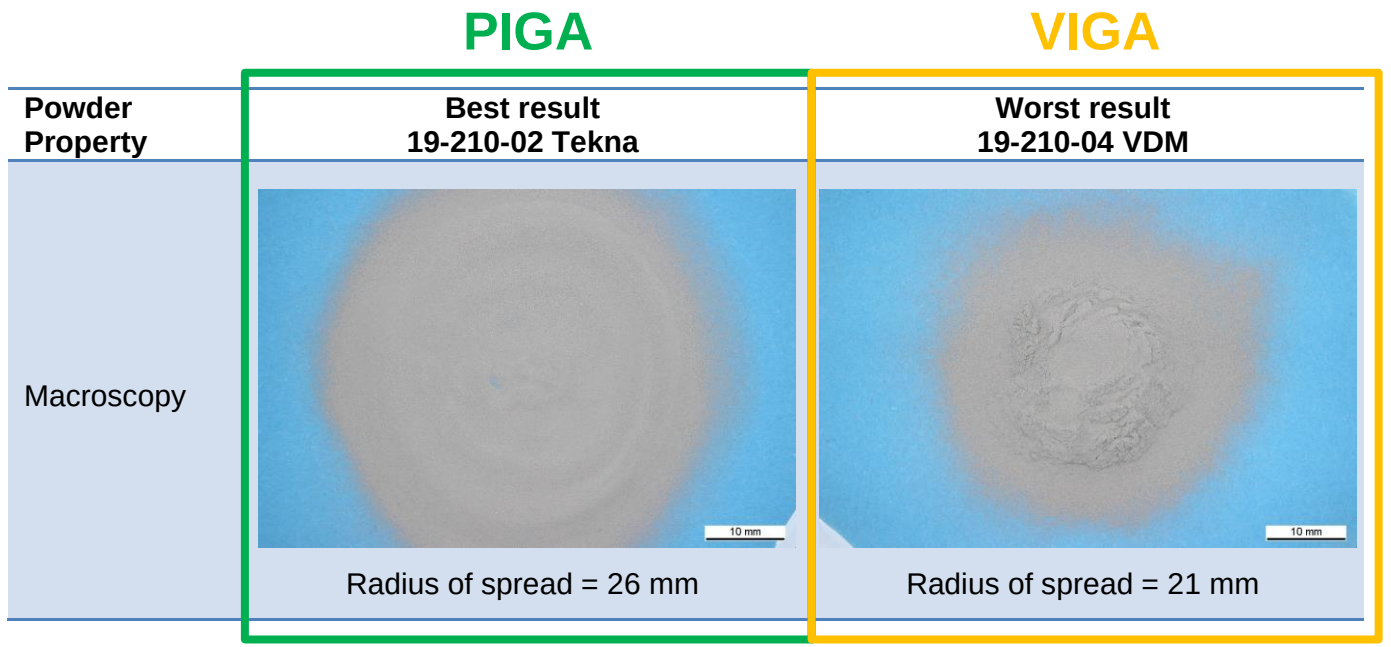
- **Characterization of powder batches by PowderGenetics**
- Template of sampling was created and used for each powder batch
- Sampling was performed according to EN ISO 3954
- Powder batches were analysed by the IABG-PowderGenetics-method
  - Macroscopic investigation
  - Chemical analysis
  - Scanning electron microscopy
  - Computed tomography

WP 3000 – Characterization of Powder Batches

- **Macroscopic investigation**

- Description of the appearance of the powder
  - homogenous grey colour
  - no abnormal colouring or obvious contamination
  - Differ in smoothness, rippleness and dispersion

- Determination of radius of spread
  - VIGA ~ 20 mm
  - PIGA ~ 25 mm



WP 3000 – Characterization of Powder Batches

- **Chemical analysis**

- IABG used CGHE, XRF and ICP-OES to determine the elemental composition

- Chemical analyses performed by the powder manufacturers and at IABG yield comparable results.

- All powder samples meet the PPS requirements

- Oxygen was analysed by Praxair and Tekna
  - IABG measured 40 % higher oxygen contents
  - Praxair: 0.013 % to 0.018 %
  - Tekna: 0.008 % to 0.011 %

Transport / Storage
due to container material

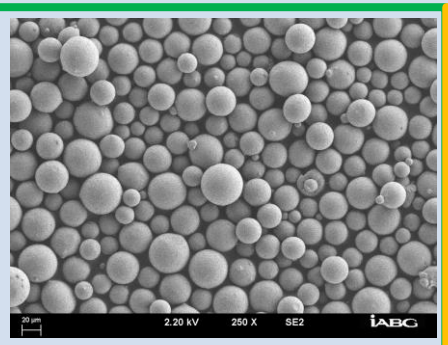
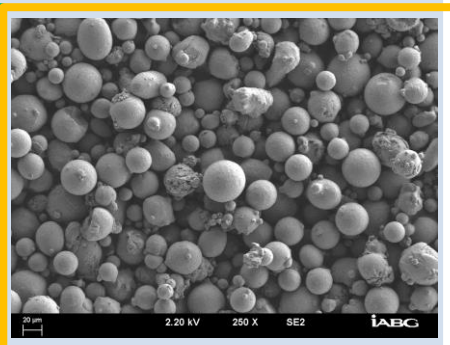
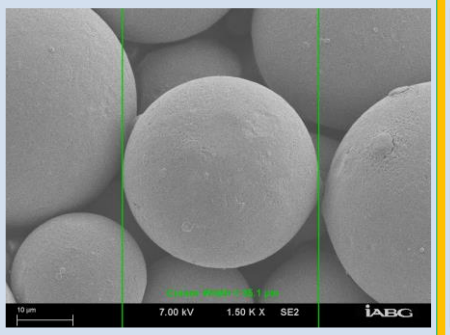
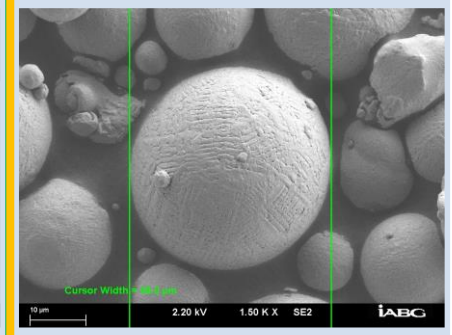
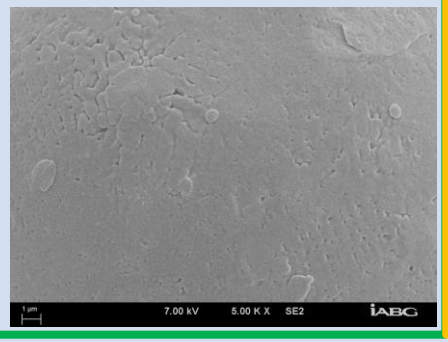
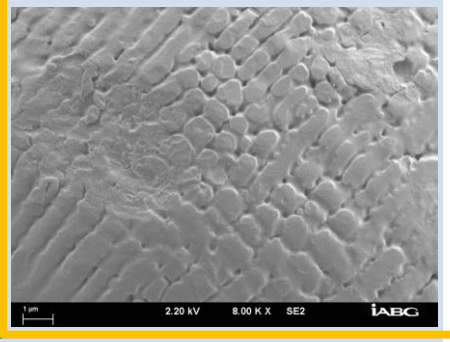
WP 3000 – Characterization of Powder Batches

- **Scanning electron microscopy**
- Description of the appearance of the powder
- Presence of non-spherical particles, satellites, shells and agglomerates
- Topography of particles at different sizes

Amount of non-spherical Particles

less → **more**

AP&C Batch 2 19-210-03-02		Praxair 19-210-01		
AP&C Batch 1 19-210-03-01	VDM Batch 2 19-210-04-02	Tekna 19-210-02	VDM Batch 3 19-210-04-03	VDM Batch 1 19-210-04-01

Powder Property	Best result 19-210-02 Tekna	Worst result 19-210-04 VDM
Scanning electron microscopy		
		
		

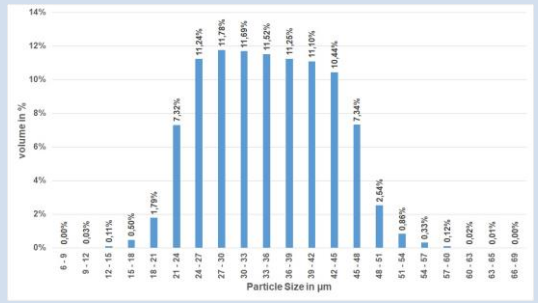
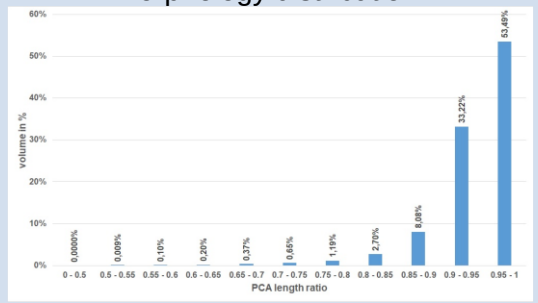
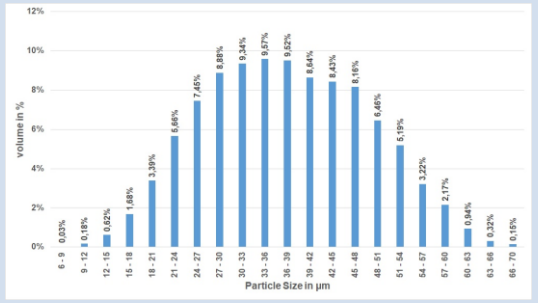
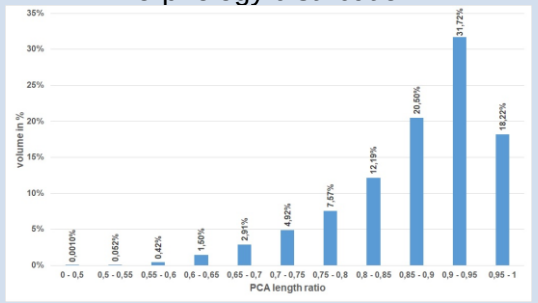
PIGA VIGA

WP 3000 – Characterization of Powder Batches

- **Computed tomography**

- Determination of:
  - Particle size
  - Particle size distribution
  - Morphology
  - Morphology distribution
  - Higher dense particles
  - Hollow spaces

- The requirements of the PPS:
  - not respected by AP&C Batch 2 and VDM powder Batch 2 and 3
  - All other powder samples comply with the particle size distribution specified.

Powder Property	Best result 19-210-02 Tekna	Worst result 19-210-04 VDM
Computed tomography	Particle size distribution  D10 = 24.1 µm D50 = 34.5 µm D90 = 45.4 µm Morphology distribution  D10 = 0.89 D50 = 0.95 D90 = 0.98	Particle size distribution  D10 = 23.3 µm D50 = 37.0 µm D90 = 52.0 µm Morphology distribution  D10 = 0.75 D50 = 0.90 D90 = 0.96
Hollow Spaces	0.006 %	0.008 %
	PIGA	**VIGA**

WP 3000 – Characterization of Powder Batches

- **Prediction**

- The results of the PowderGenetics investigation led to the following predictions:

Specimen property	Best result	2nd best result	3rd best result	Worst result
Processability	Tekna (PIGA)	AP&C (PIGA)	VDM (VIGA)	Praxair (VIGA)
Roughness - R_a	Praxair (VIGA)	AP&C (PIGA)	Tekna (PIGA)	VDM (VIGA)
Roughness - R_z	AP&C (PIGA)	Tekna (PIGA)	Praxair (VIGA)	VDM (VIGA)
Pore content	Tekna (PIGA)	AP&C (PIGA)	VDM (VIGA)	Praxair (VIGA)
Tensile strength	Depends on the respective specimen position in the reaction chamber			

PIGA **VIGA**

WP 4000 – Production of test specimen

Manufacturing process

Final setup for all specimens used at EOS M290:

Process Parameter	Description
Laser power	285 W
Scan speed	960 mm / s
Lane spacing	0.11 mm
Layer thickness	0.04 mm
Built plate	30 mm steal plate (C45)
Built plate temperature	80 °C
Recoater	Build job # 1 to 3: HSS Blade Build job # 4 to 8: Carbon Brush
Shielding gas	Argon O_2 < 0.1 % (measured) N_2 < 0.4 % (predicted)
Laser strategy	1. Hatch (285 W, 960 mm / s) 2. First Contour (138 W, 390 mm / s) 3. Second Contour (80 W, 800 mm / s)
Change of the scanning orientation between two consecutive layers	67 °

WP 4000 – Production of test specimen

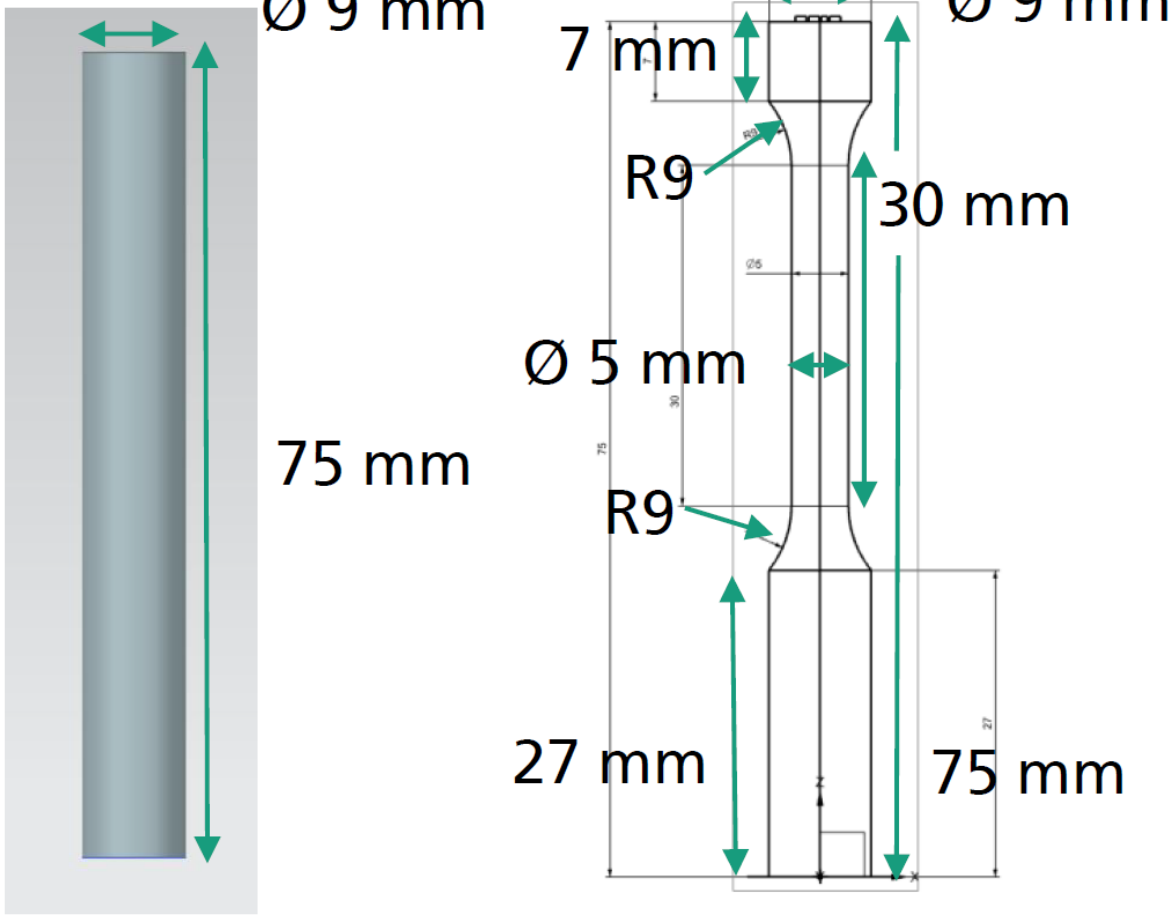
- **Specimen geometry**

- A) Cylinders (even numbers)
  diameter of 9 mm and length of 75 mm
  Heat treatment and machining

- B) Cylinders (uneven numbers)
  diameter of 9 mm and length of 75 mm
  Machining

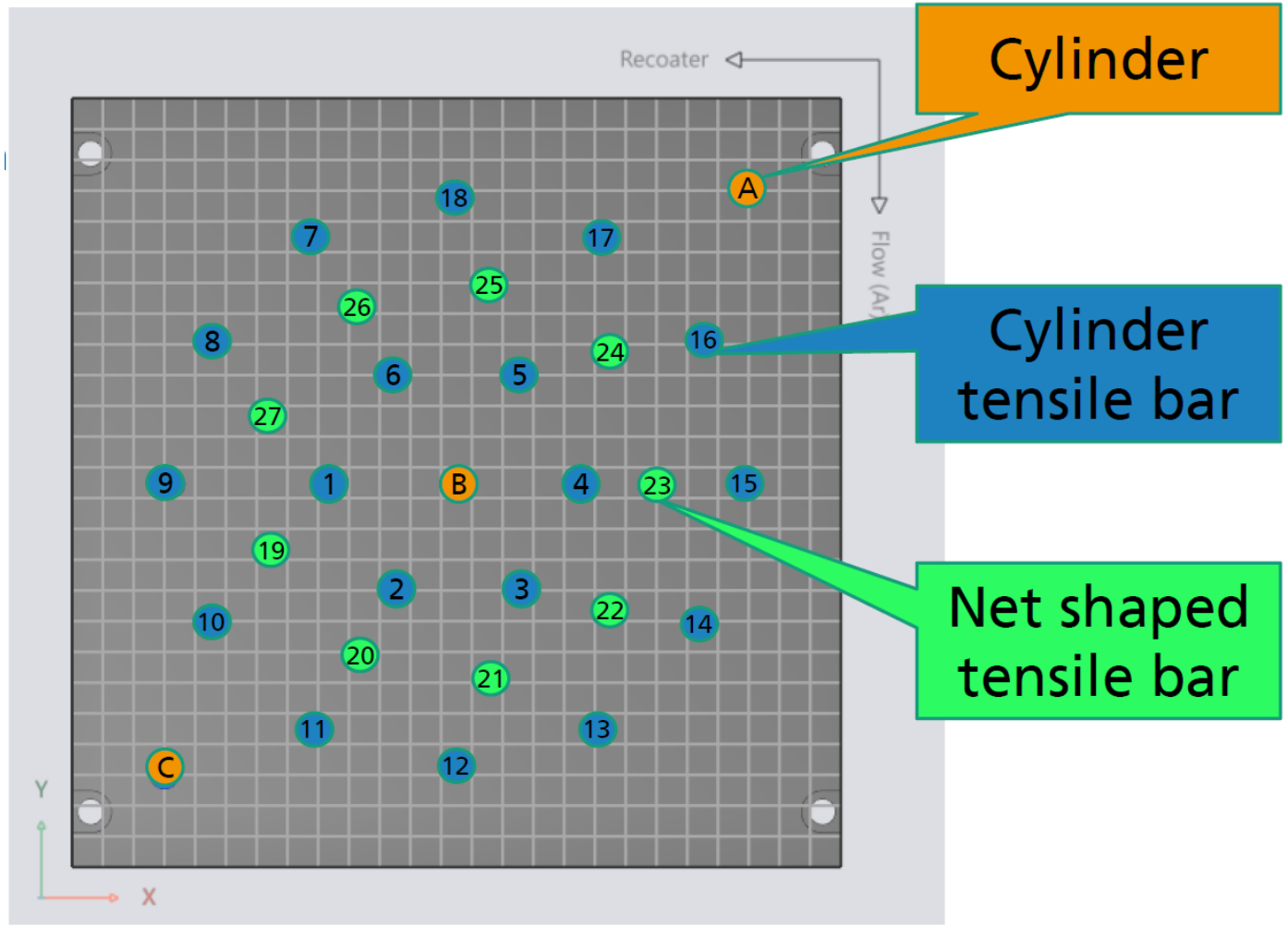
- C) Net shaped
  B 5 x 50 tensile strength specimens
  no post-processing

- Reference cylinder tensile bar



WP 4000 – Production of test specimen

- Manufacturing templates were created and filled-in for each build job



WP 4000 – Production of test specimen

Fraunhofer-Einrichtung für Gießerei-, Composite- und Verarbeitungstechnik IGCV

Build job number	Start Date of additive manufacturing	Powder used	Interruption of build job	Remarks
1	26.08.2020	19-210-01 Praxair (VIGA) usecycle #1	Yes	Recoater contact with 3 specimens
2	07.09.2020	19-210-01 Praxair (VIGA) usecycle #2	No	Specimens are not used for the following WP
3	08.09.2020	19-210-02 Tekna (PIGA) usecycle #1	Yes	Recoater contact with one specimen
4	15.09.2020	19-210-03 AP&C (PIGA)	No	Specimens are used for the following WP
5	16.09.2020	19-210-04 VDM (VIGA)	No	Specimens are used for the following WP
6	17.09.2020	19-210-02 Tekna (PIGA) usecycle #2	No	Specimens are used for the following WP
7	18.09.2020	19-210-01 Praxair (VIGA) usecycle #3	Yes	Pressure drop; due to automated filter cleaning process
8	21.09.2020	19-210-01 Praxair (VIGA) usecycle #4	No	Specimens are used for the following WP

WP 4000 – Production of test specimen

- **Praxair**

- **Received Praxair specimen by IGCV**

- **Received Praxair specimen by IGCV**

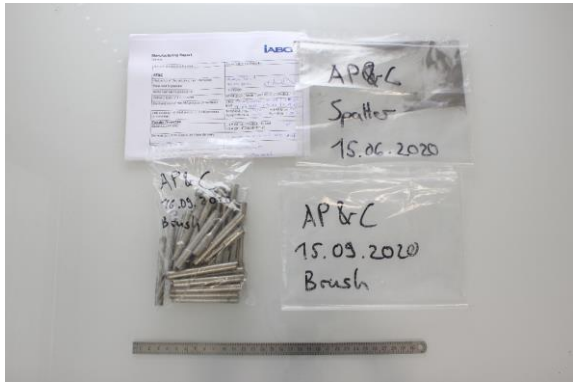
- **Tekna**

- **Received Tekna specimen by IGCV**

- **Received Tekna specimen by IGCV**

WP 4000 – Production of test specimen

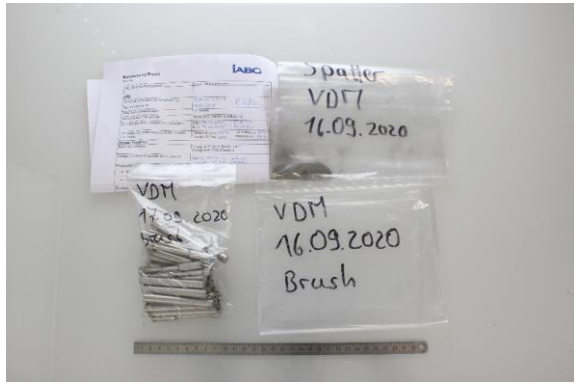
- **AP&C**



- **Received AP&C specimen by IGCV**

- **Received AP&C specimen by IGCV**

- **VDM**



- **Received VDM specimen by IGCV**

- **Received VDM specimen by IGCV**

WP 5000 – Analysis as built

- **Density measurement**

- All specimens are in the range of 99.5 % of the theoretical density of 8.2 g / cm³

IBL-M Sample No.	Mean value ρ in g / cm³
19-210-01 Praxair (VIGA)	8.22
19-210-02 Tekna (PIGA)	8.17
19-210-03 AP&C (PIGA)	8.17
19-210-04 VDM (VIGA)	8.21

WP 5000 – Analysis as built

Roughness measurement

- All specimens have a comparable roughness

Specimen property	Best result	2nd best result	3rd best result	Worst result
Roughness - R_a predicted	Praxair (VIGA)	AP&C (PIGA)	Tekna (PIGA)	VDM (VIGA)
measured	Praxair 4.6 µm	AP&C 5.2 µm	Tekna 5.3 µm	VDM 5.7 µm
Roughness - R_z predicted	AP&C (PIGA)	Tekna (PIGA)	Praxair (VIGA)	VDM (VIGA)
measured	Praxair 35.8 µm	AP&C 39.0 µm	Tekna 40.0 µm	VDM 41.3 µm
D50 particle size of the powder used	Praxair 31.3 µm	AP&C 32.2 µm	Tekna 34.5 µm	VDM 37.0 µm

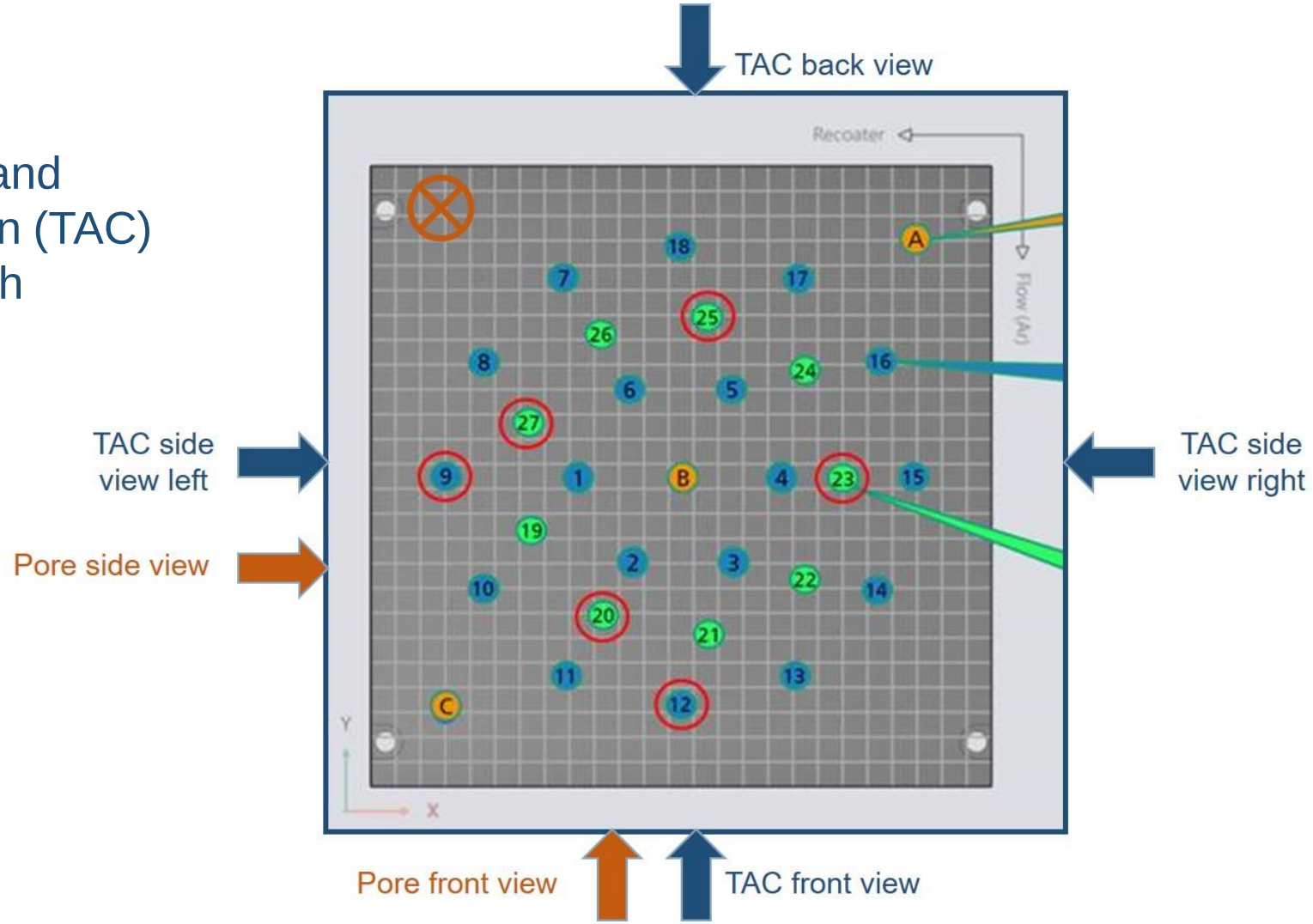
- Prediction of R_a values was correct

WP 5000 – Analysis as built

- **Computed tomography**

- porosity analysis (Pore) and target / actual comparison (TAC) have been performed with 10 microns resolution

- 24 CT-scans (6 per powder sample)



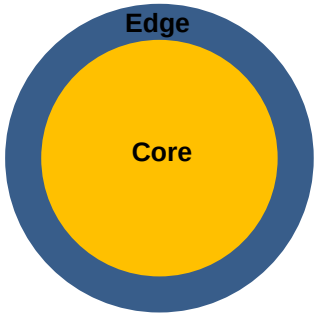
WP 5000 – Analysis as built

- **Computed tomography**

- porosity analysis

IBL-M Sample No.	Edge porosity Mean value in %	Core porosity Mean value in %	Predictions
19-210-01 Praxair (VIGA)	0.034	< 0.0001	Worst result
19-210-02 Tekna (PIGA)	0.027	0.0002	Best result
19-210-03 AP&C (PIGA)	0.032	0.0001	2nd best result
19-210-04 VDM (VIGA)	0.032	< 0.0001	3rd best result

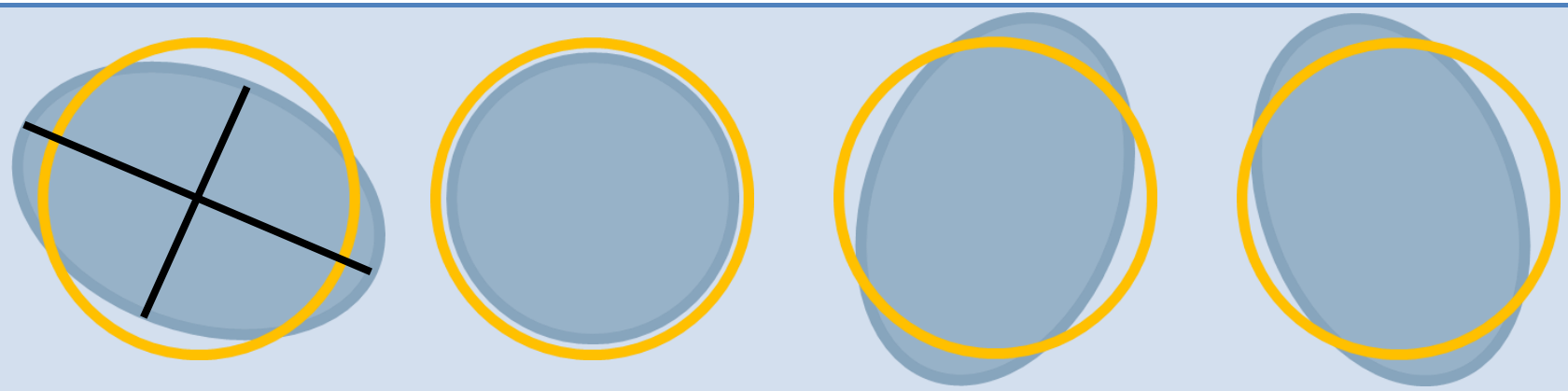
- Results of the edge porosity corresponds to the prediction



WP 5000 – Analysis as built

- **Computed tomography**

- target / actual comparison

Shape of test area	Specimen #20	Specimen #23	Specimen #25	Specimen #27
Top view				
Description	Orange represents the target geometry (ideal cylindrical) Blue represents the actually generated geometry in the cross-section.			

- Difference between main to secondary axis: 10 x R_a (5 µm) = 50 µm
  or in absolute values: 4860 µm to 4910 µm

WP 6000 – Post-processing of test specimen

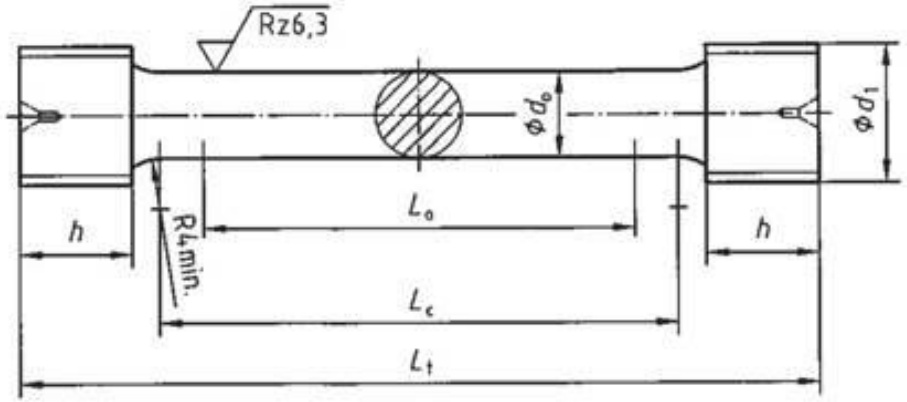
- **Heat treatment**

- Heat treatment process for condition B:
  - vacuum oven at 650 °C
  - Holding time of 1 hour

- **Machining**

- Condition A and B specimens were machined to round tensile specimens with the geometry B 5 x 50

- M8 threads were machined into all specimens



WP 7000 – Analysis after post processing

- **Roughness measurement**

- All machined specimens (condition A and B) show comparable roughness values

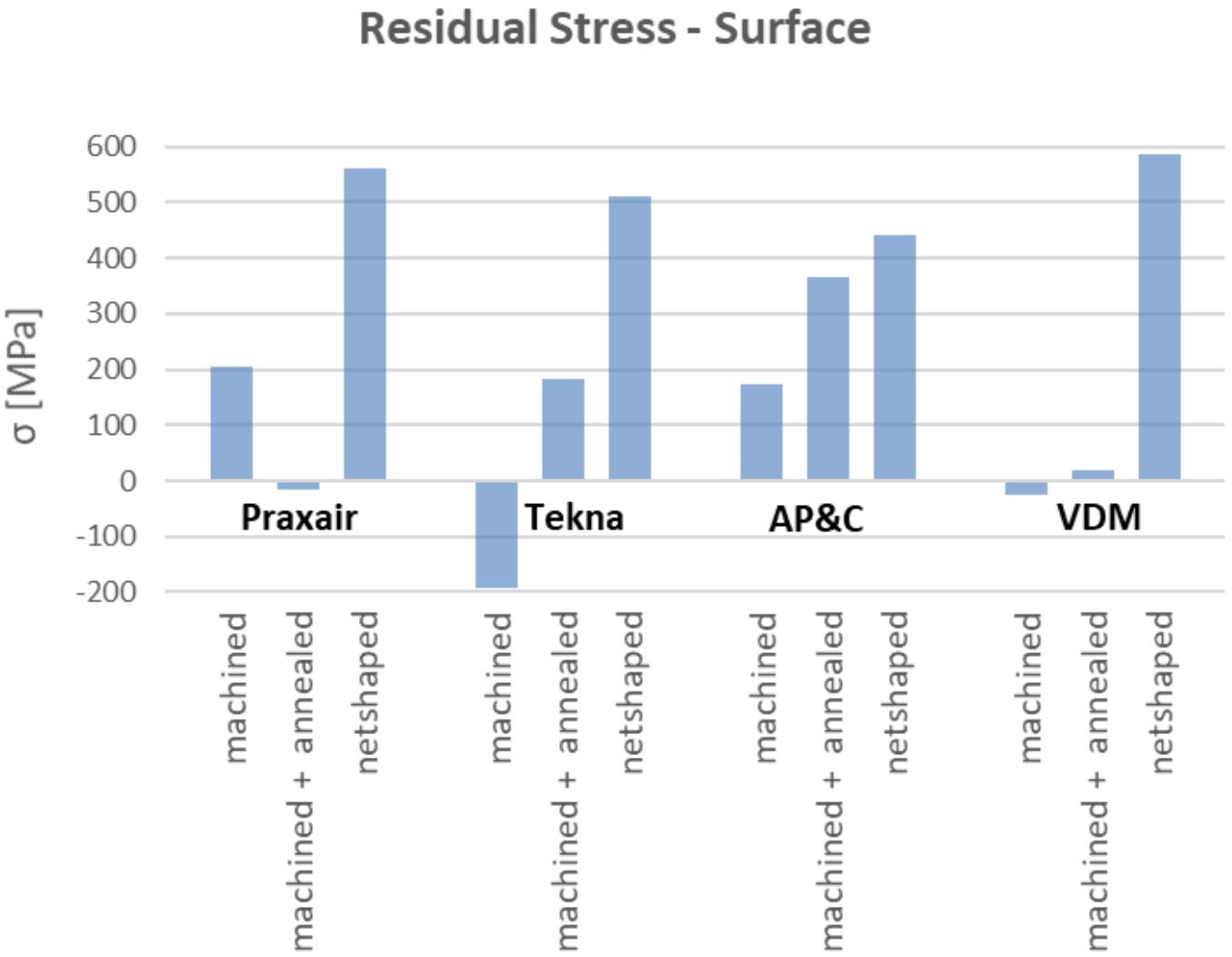
IBL-M Sample No.	R_a in µm	R_z in µm	R_{z1max} in µm
19-210-01 Praxair (VIGA)	0.6	3.5	4.3
19-210-02 Tekna (PIGA)	0.6	3.5	4.5
19-210-03 AP&C (PIGA)	0.7	3.7	4.8
19-210-04 VDM (VIGA)	0.7	3.6	4.4

WP 7000 – Analysis after post processing

- **Residual stress measurement**

- Net shaped specimens (condition C) contain tensile stress on the surface with similar values.

- Surface stresses (condition A and B) differ between compressive and tensile stresses after machining



WP 7000 – Analysis after post processing

- **Residual stress measurement – Depth profile for VDM specimen**

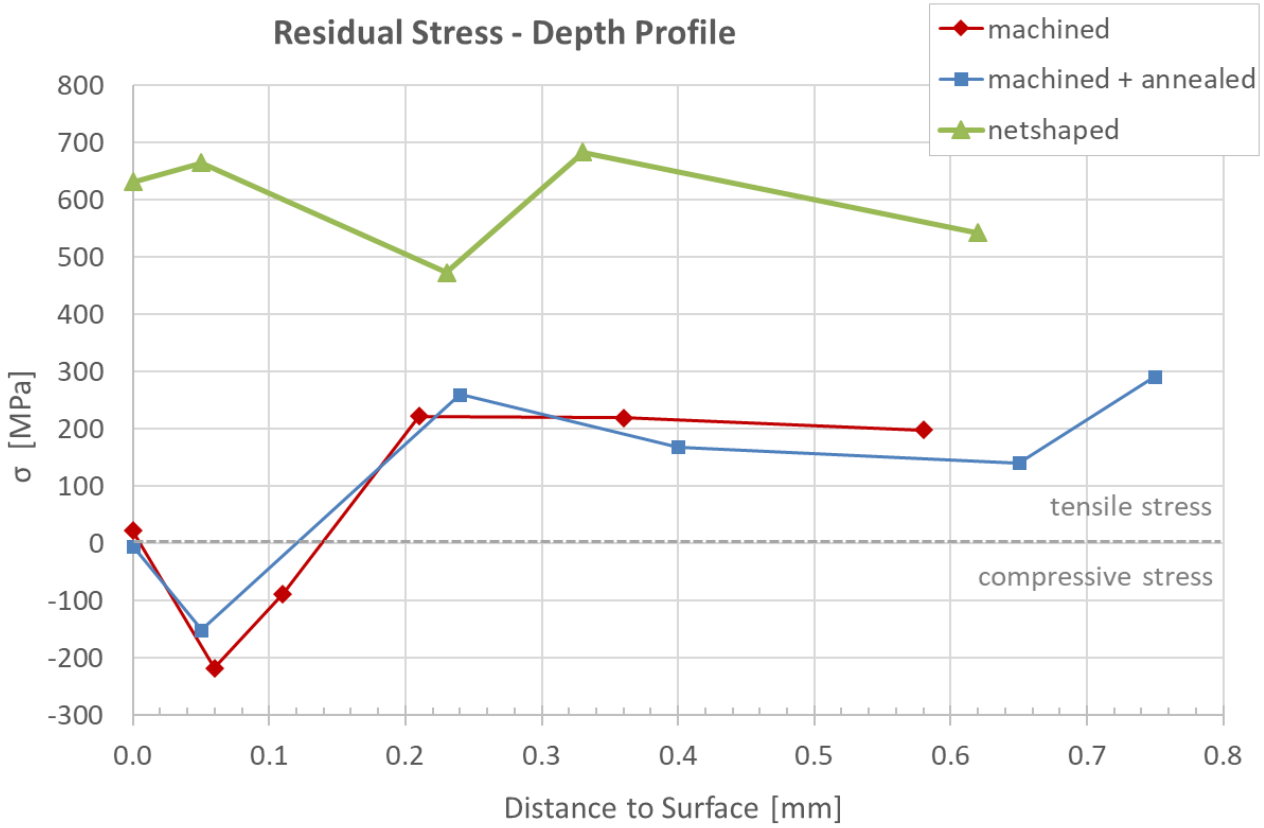
- Condition C:
  constant residual stresses
  to a depth of 0.6 mm

- Condition A and B have similar values

  surface starts with a stress free state

  compressive stress
  to a depth of 0.15 mm

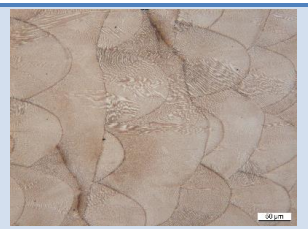
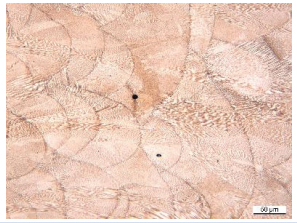
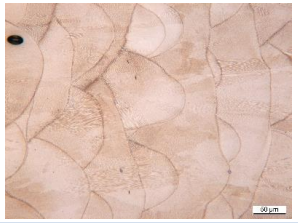
  followed by tensile stress
  to the last measuring point



WP 7000 – Analysis after post processing

- **Metallographic investigation**

- Light microscopical images of transverse and longitudinal sections:

Condition	19-210-01 Praxair - VIGA	19-210-02 Tekna - PIGA	19-210-03 AP&C - PIGA	19-210-04 VDM - VIGA
A Longi-tudinal sections				
B Longi-tudinal sections				
C Longi-tudinal sections				

- Microstructure between conditions and samples is similar

WP 7000 – Analysis after post processing

- **Hardness test by Vickers**

- Condition A (machined) has the highest hardness values followed by condition B (machined and annealed) and condition C (net-shaped)

- tensile strength was converted from the hardness values

Parameter / Specimen	19-210-01 Praxair - VIGA	19-210-02 Tekna - PIGA	19-210-03 AP&C - PIGA	19-210-04 VDM - VIGA
Condition A Mean value in HV1	**391**	**386**	**381**	**393**
Respective tensile strength in MPa	1254	1238	1222	1260
Condition B Mean Value in HV1	**313**	**316**	**311**	**308**
Respective tensile strength in MPa	1004	1013	997	988
Condition C Mean Value in HV1	**306**	**302**	**306**	**309**
Respective tensile strength in MPa	981	968	981	991

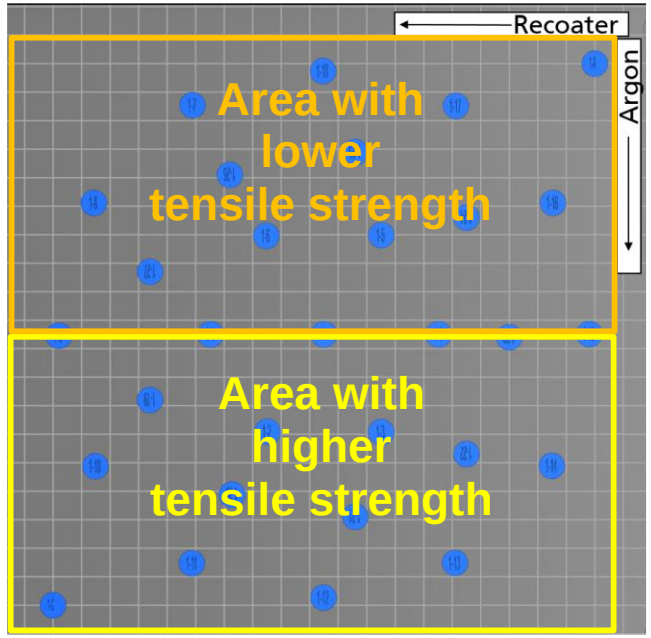
Condition	mean grain diameter in µm
Condition A machined	25.3
Condition B machined and annealed	28.9
Condition C net shaped	27.7

WP 8000 – Tensile strength tests

- Condition A (machined) has the highest tensile strength values followed by condition B (machined and annealed) and condition C (net-shaped)

- Depends on position of the specimen

Specimen property	Best result	2nd best result	3rd best result	Worst result
Predicted Processability	Tekna (PIGA)	AP&C (PIGA)	Praxair (VIGA)	VDM (VIGA)
Predicted Tensile strength	Depends on the respective specimen position in the reaction chamber			
Condition A R_m measured	Tekna 1143 MPa	AP&C 1138 MPa	Praxair 1138 MPa	VDM 1123 MPa
Condition B R_m measured	Tekna 960 MPa	AP&C 953 MPa	Praxair 950 MPa	VDM 945 MPa
Condition C R_m measured	Tekna 942 MPa	Praxair 930 MPa	AP&C 929 MPa	VDM 926 MPa



- Results of the tensile strength corresponds to the prediction

Conclusion

PowderGenetics describes the elemental powder properties at the highest possible information depth.

► **holistic powder characterization**

Determination of powder processability by evaluation and correlation of qualitative and quantitative information of powder properties.

► **powder processability**

Detection of smallest deviations and the evaluation of their effect on both the manufacturing process and the correlated part quality.

► **process stability**

IABG successfully established a correlation between IN718 powder properties and the resulting part quality

► **prediction of part quality before printing**

Outlook

Follow-on

DeRisk

Prediction

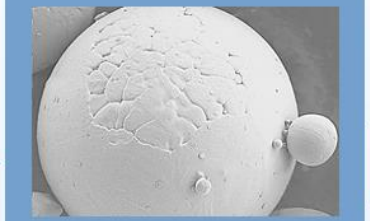
Supply Chain

Influence of filling, bottling & transportation

Procurement

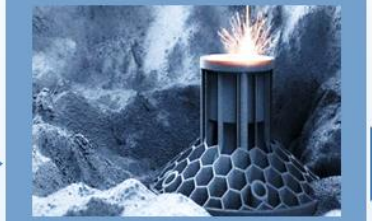
Powder survey
Procurement specs
Powder Procurement

Powder Analysis



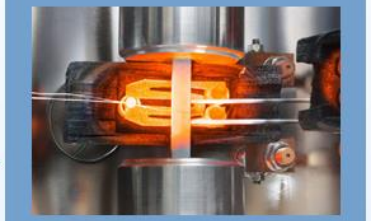
PowderGenetics
Elemental powder characteristics

AM-Process



Sample manufacturing
"Frozen" process

Sample Quality



Material characterization
Static properties

Correlation

Statistic Add On

Additional manufacturers for increased survey & statistics

Correlation Add On

Correlation platform based on artificial intelligence

Properties Add On

LT & HT static
Creep
Fatigue

Your Contact

Marion Eiber
Head of Department
Material- and
Failure Analyses

Tel: +49 89 6088-2441
Email: eiber@iabg.de

Tom Näke
Project Leader
Material- and
Failure Analyses

Tel: +49 89 6088-4153
Email: naeke@iabg.de

IABG mbH
Einsteinstrasse 20
85521 Ottobrunn
Germany