Additive Manufacturing – versatile but challenging process

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Fabrication of three dimensional objects

Additive Manufacturing, Rapid Prototyping, Free Form Fabrication, Laser 3D Structuring ……..

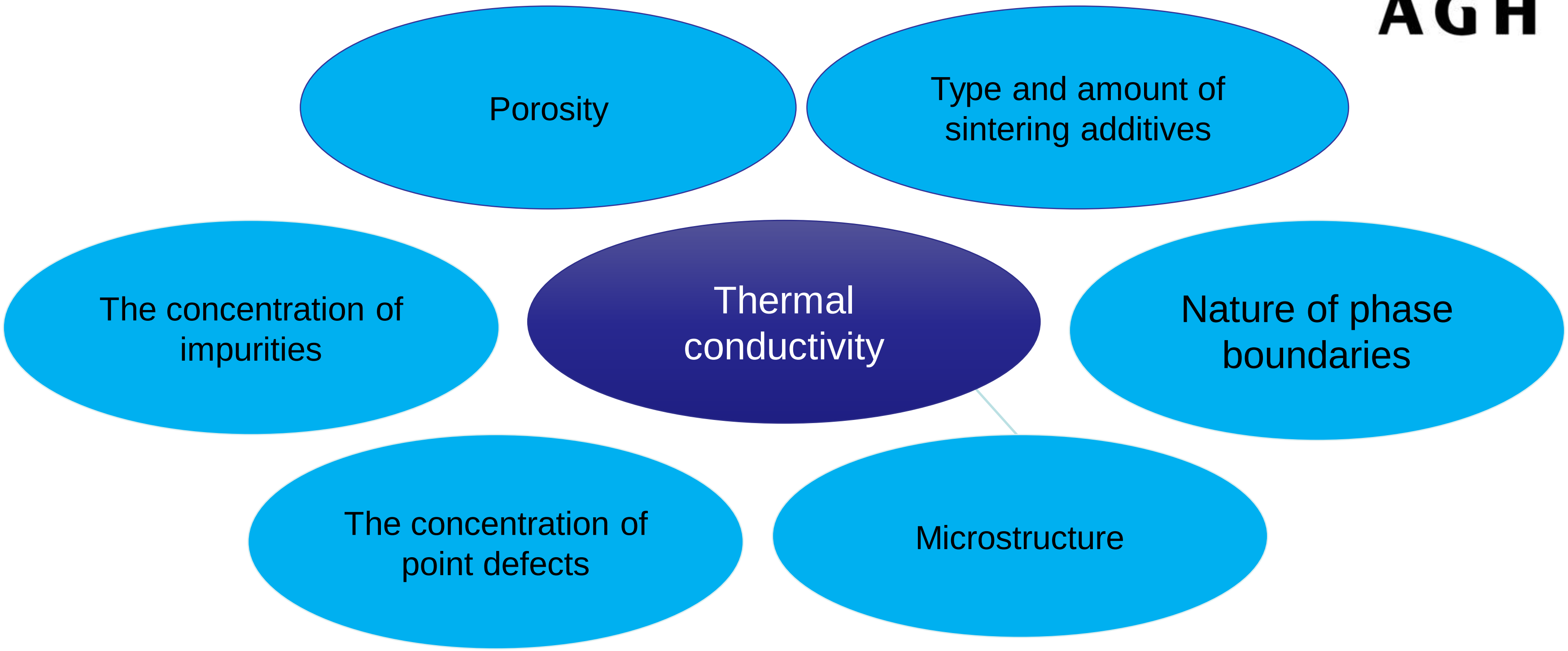
Additive Manufacturing (bottom-up approach)

	Additive Manufacturing	Physico-chemical processes
1)	Fused Deposition Modeling (FDM) (Polimers)	metling and UV-curring
2)	Stereolitography (SLA) Digital Light Processing (DLP)	UV-curring
3)	Laminated Object Manufacturing (LOM) (celuloze polimers)	Thermolisys and hardening polimers
4)	3D Colour Jet Printing (3CJP) (gypsum)	Hydration reaction of gypsum
5)	Selective Laser Sintering (SLS) (ceramic and metallic powders)	Sintering
6)	Laser Engineering Net Shaping (LENS) (ceramic and metallic powders)	Sintering
7)	„Clading" Laser Metal Deposition LMD (metallic powders)	Metllting and crystallization

Laser Processing Top-down approach

Additive Manufacturing	Physico-chemical processes
Laser Ablation	Sublimation and evaporation of ceramic and
Laser cutting, Scratching, Drilling	Thermall dyssociation evaporation

High thermal conductivity materials
microstructural aspects:

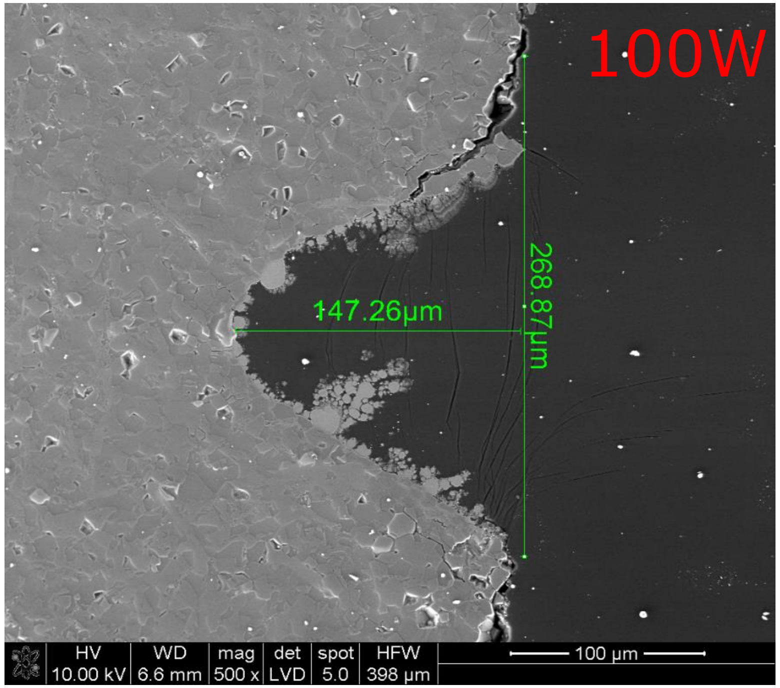


Aluminum nitride is a material of great application potential:
thermal conductivity of about 260W/mK,
broadband insulator 6,2 eV
piezoelectric properties, the thermal expansion coefficient close to silicon

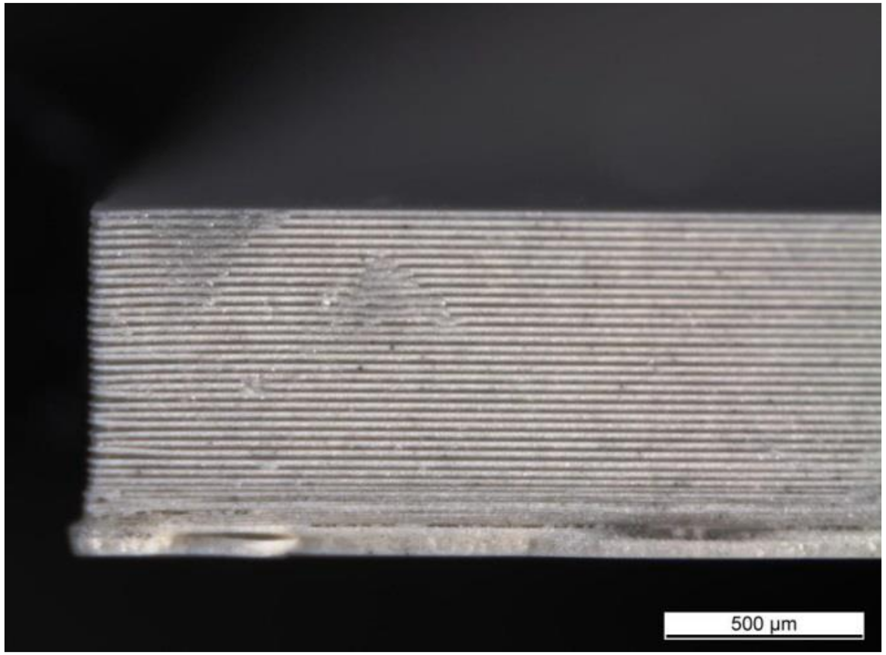
Diamond is the material of the future:
thermal conductivity of about 2000W/mK,
electrical insulator, hardness 36 GPa,
As an additive to composites it can replace the tungsten carbide used in cutting tools

3D Shaping of AlN micro heat exchanger
a) Laser processing; b) Digital light processing

a)



b)

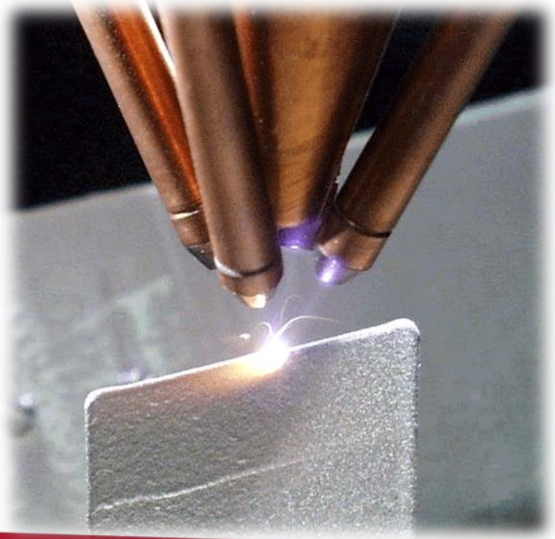


Aluminum nitride (AlN) offers remarkable thermal conductivity which makes it suitable candidate in manufacturing of high-tech heat exchangers. The present work aims to 3D shaping of a micro-sized heat exchanger made of AlN.

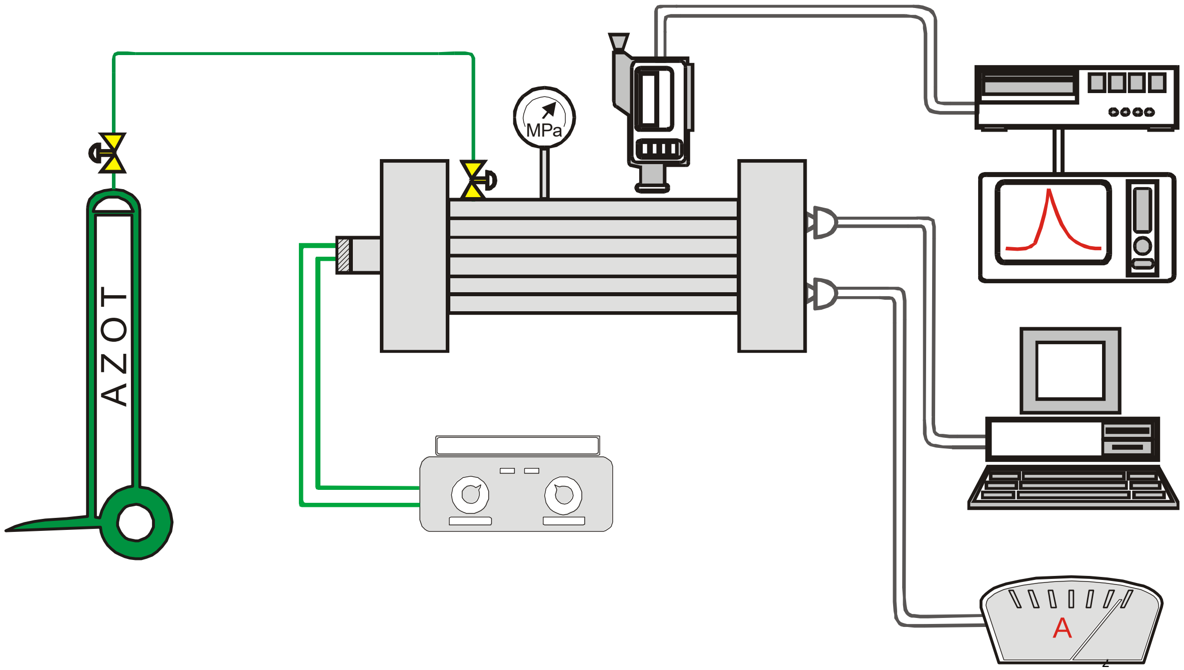
Paulina Ożóg PhD thesis „Shaping of AlN powders by Additive Manufacturing applying UV-curable dispersions" AGH and EMPA 2019 supervisors: Prof. D. Kata, Prof. T. Graule.

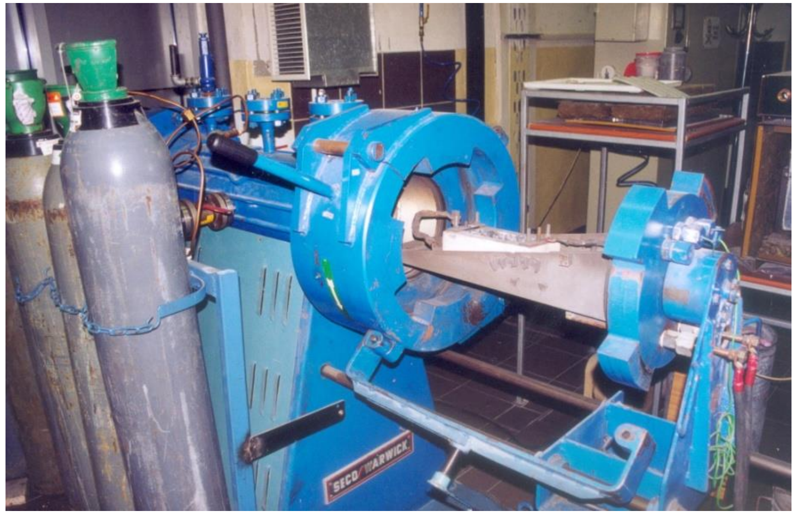
Additive Manufacturing of AlN and AlN-GPLs composites

1. Hybrid apparatus for laser cladding, surface ablation, welding, cutting and SHS reactions
2. JK2000FL equipped with ytterbium doped wire fiber
3. Laser beam with wavelength of 1063[nm] ± 10[nm]



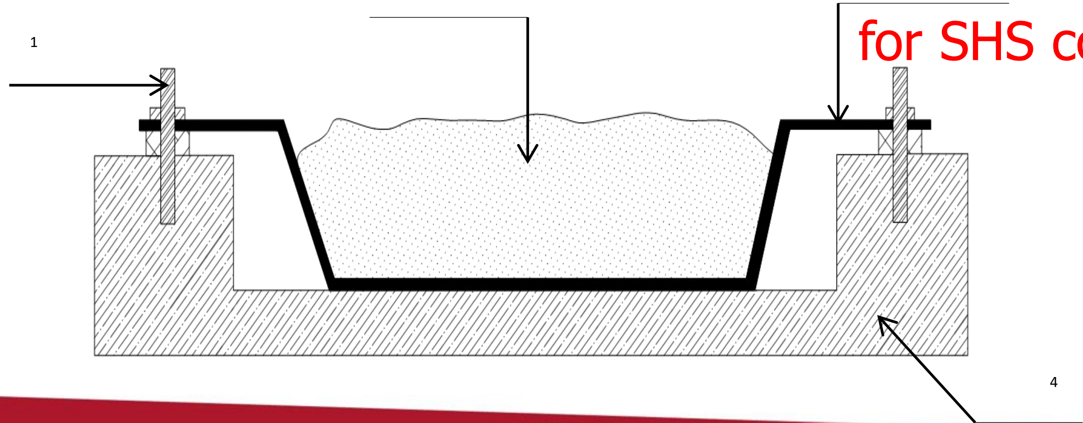
Synthesis of Aluminium nitride by SHS

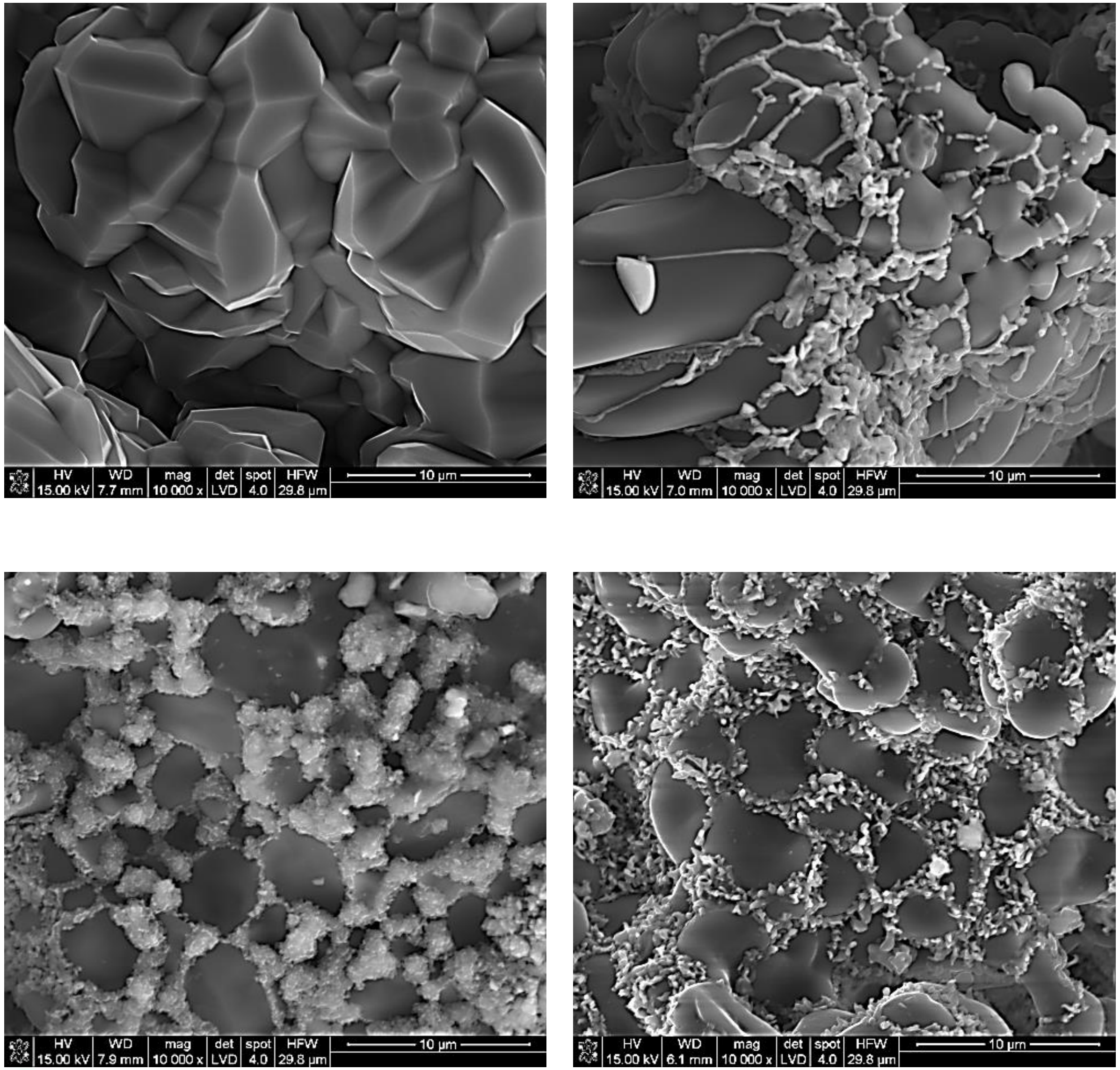




1) Heating supply
2) Mixture of (Al+ AlN)
3) Powdery bed
4) Heat insulator

Scheme of SHS equipment for SHS combustion





Morphology of SHS derived AlN powders:

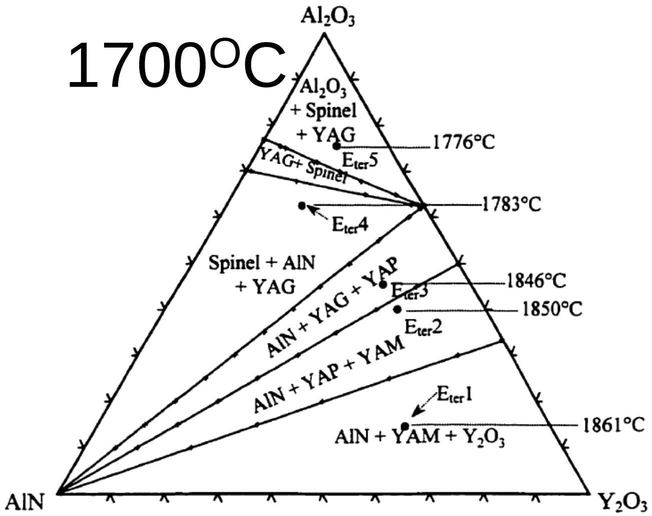
a) Pure AlN

b) AlN + 6% Y_2O_3;

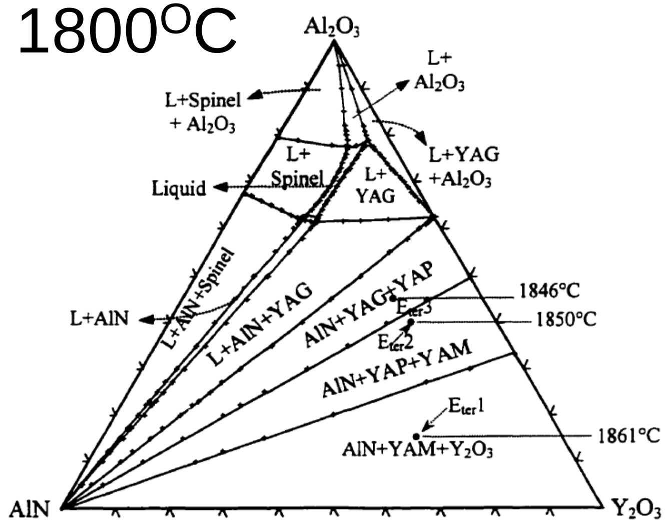
c) AlN + 8% Y_2O_3;

d) AlN +10% Y_2O_3;

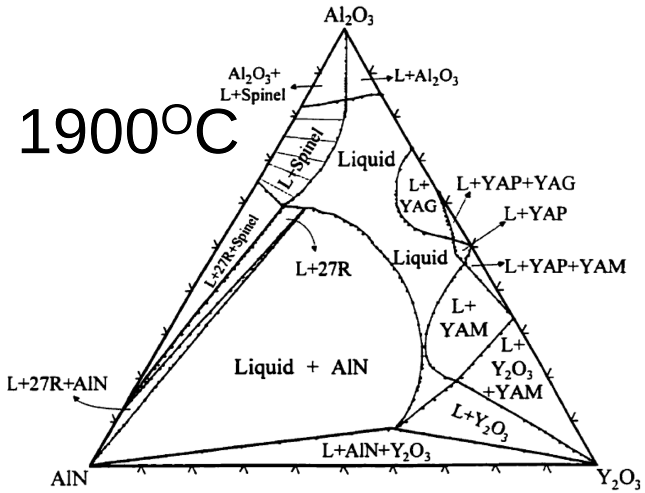
1700^{O}C



1800^{O}C



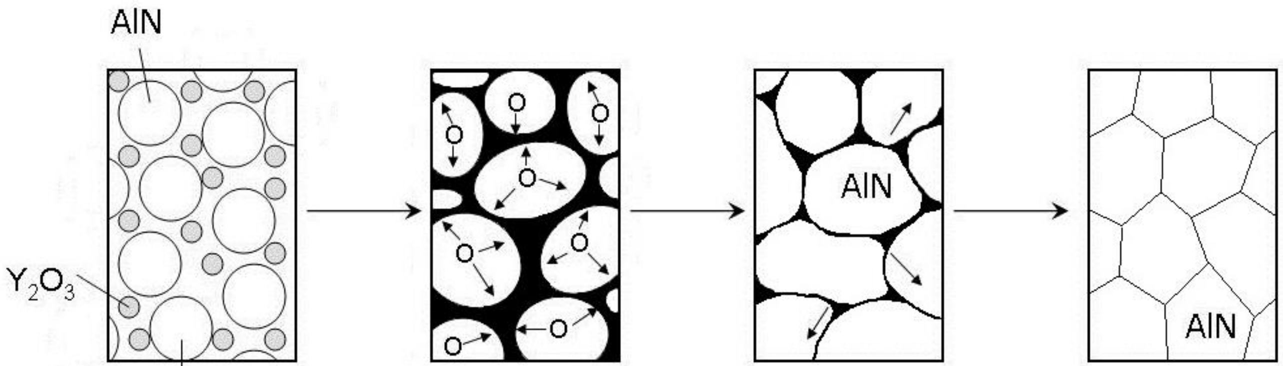
1900^{O}C

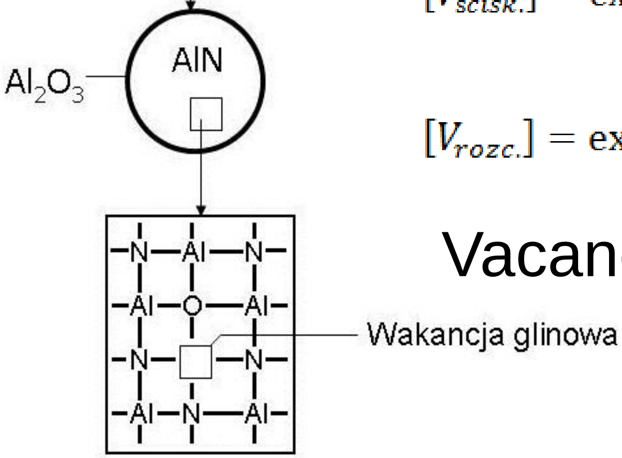


Phase Diagram of AlN-Y_2O_3-Al_2O_3 system

Mamoun Medraj, PHASE EQUILIBRIA IN THE AlN-Al203-Y203 SYSTEM - *UTILITY IN AIN PROCESSING*, McGill University Montreal Canada, PhD, 2001

Sintering of AlN with addition of Y_2O_3

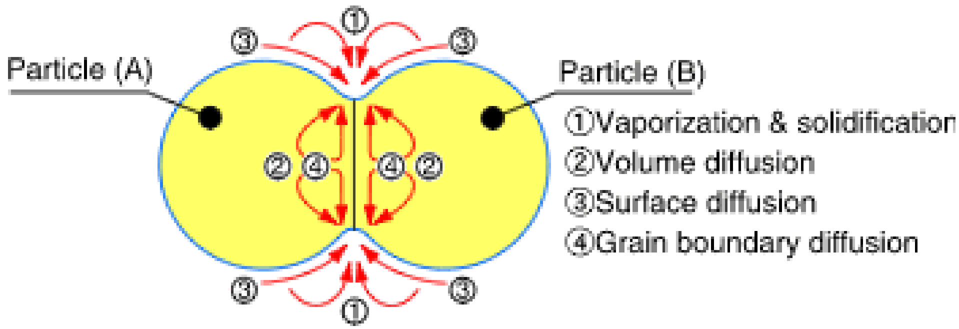




$$[V_{ścisk.}] = \exp\left(-\frac{E_v + \sigma\Omega_v}{kT}\right) = [V_0]\exp\left(-\frac{\sigma\Omega_v}{kT}\right)$$ → compressive stress

$$[V_{rozc.}] = \exp\left(-\frac{E_v - \sigma\Omega_v}{kT}\right) = [V_0]\exp\left(\frac{\sigma\Omega_v}{kT}\right)$$ → tensile stress

Vacancies Al



Stress at the grain boundary is not enough for purification of AlN grains during sintering

Grain boundary reactions during AlN sintering

$3Y_2O_3 + 5Al_2O_3 \rightarrow 2Y_3Al_5O_{12}$ (i) YAG

$5Y_2O_3 + 5Al_2O_3 \rightarrow 10YAlO_3$ (ii) YAP

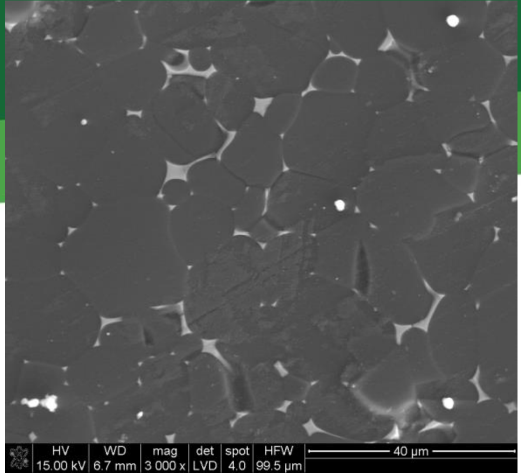
$10Y_2O_3 + 5Al_2O_3 \rightarrow 5Y_4Al_2O_9$ (iii) YAM

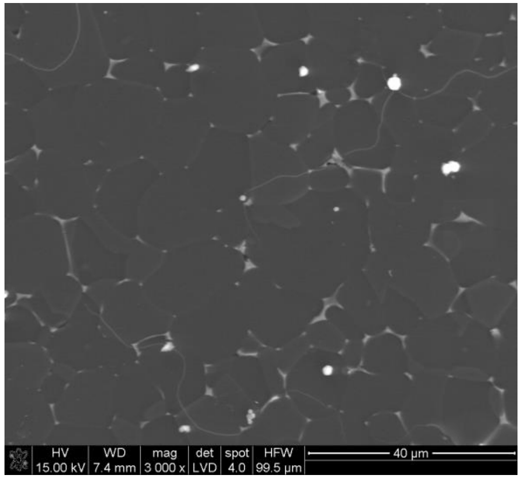
$2Y_3Al_5O_{12} + 2Y_2O_3 \rightarrow 10YAlO_3$ (iv)

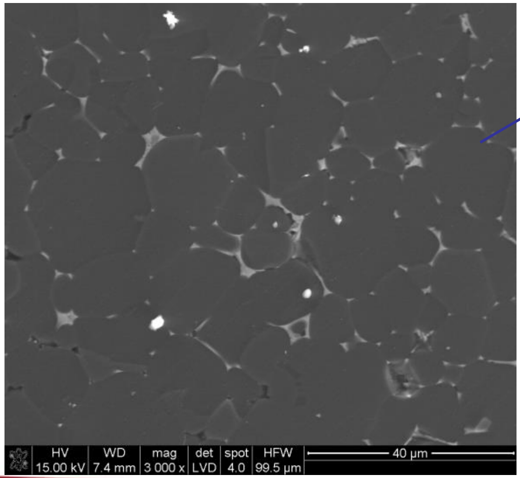
$10YAlO_3 + 5Y_2O_3 \rightarrow 5Y_4Al_2O_9$ (v)

Phase	Formula	Ratio Y_2O_3-Al_2O_3	Crystallographic system	λ [W/mK]*
YAG	$Y_3Al_5O_9$	3:5	Regular	2,5
YAP	$YAlO_3$	1:1	Orthorhombic, hexagonal	4,3
YAM	$Y_4Al_2O_9$	2:1	Monoclinic	7,4

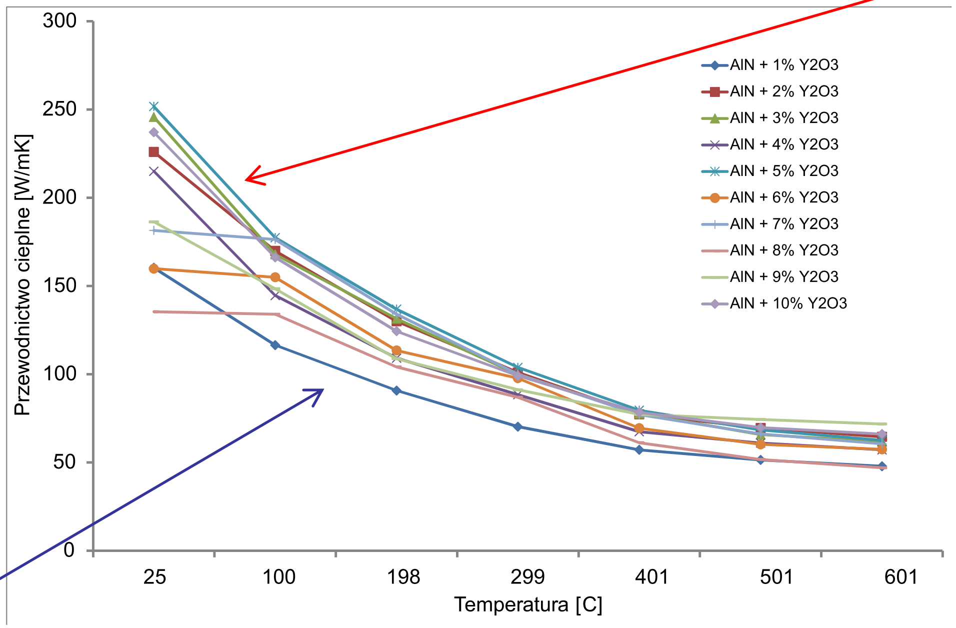
*T.B. Jackson, A.V. Virkar, K.L. More, R.B. Dinwiddie, and R.A. Cutler, J. Am. Ceram. Soc. 80, 1421 (1997).

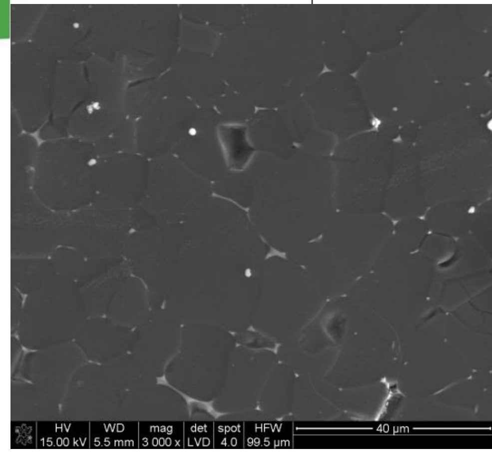


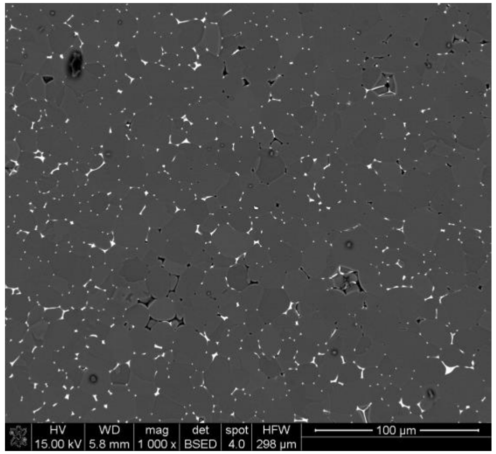


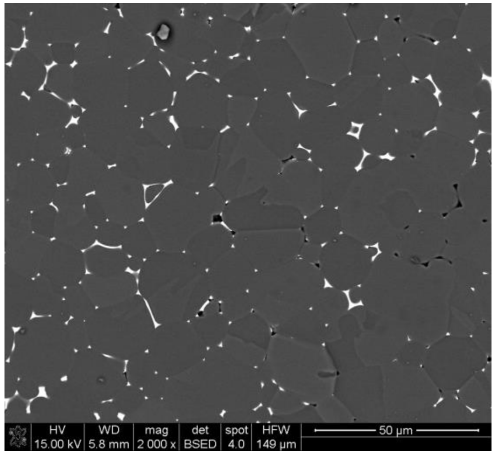


Pressureless sintering 1900ºC 1,0 hour

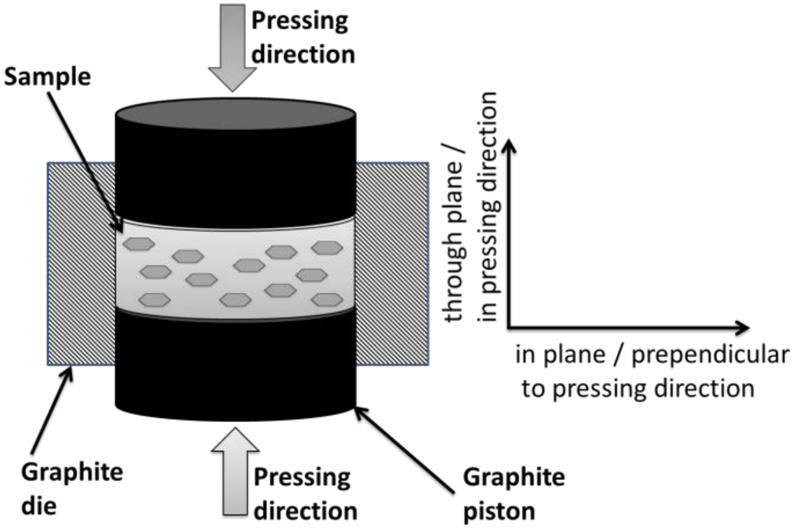


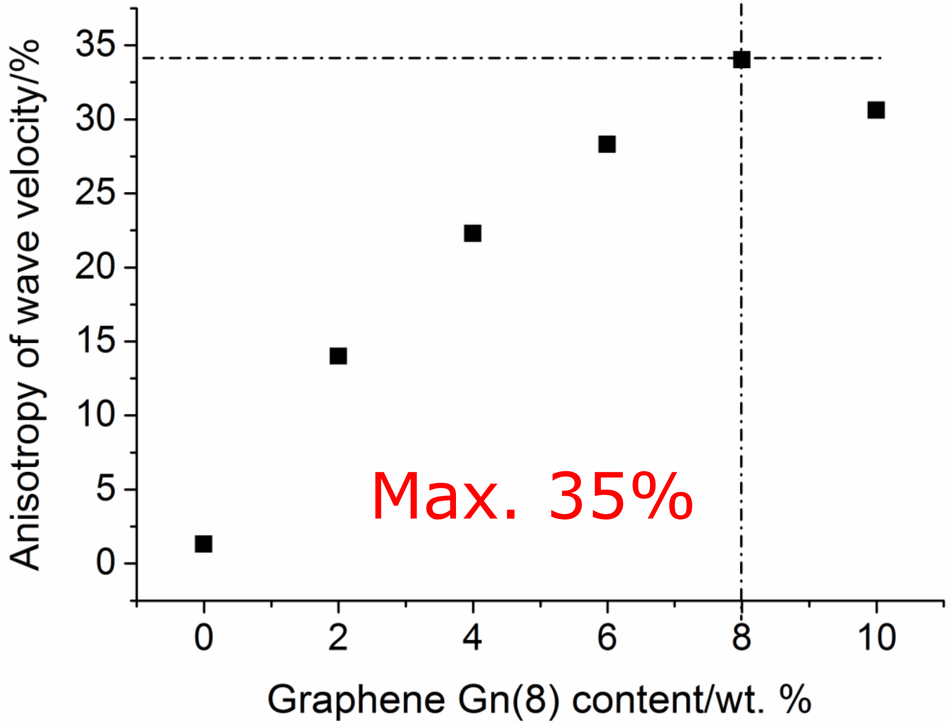






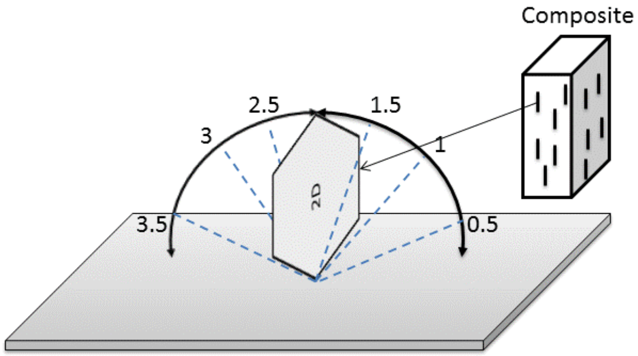
Anisotropy of AlN/GPLs composites (ultrasonic measurements)



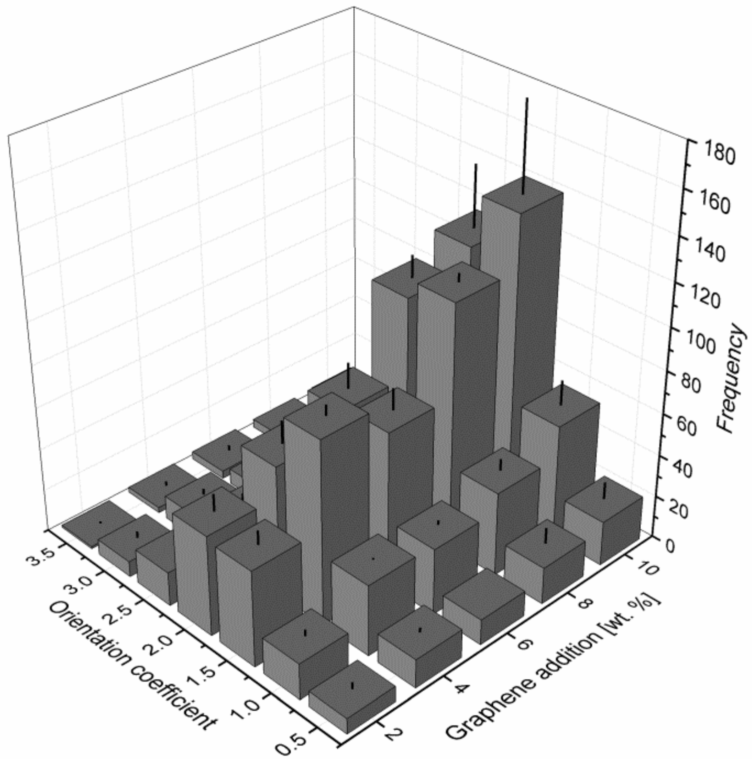


Anisotropy of AlN/GPLs composites (Computer-aided microstructure analysis)

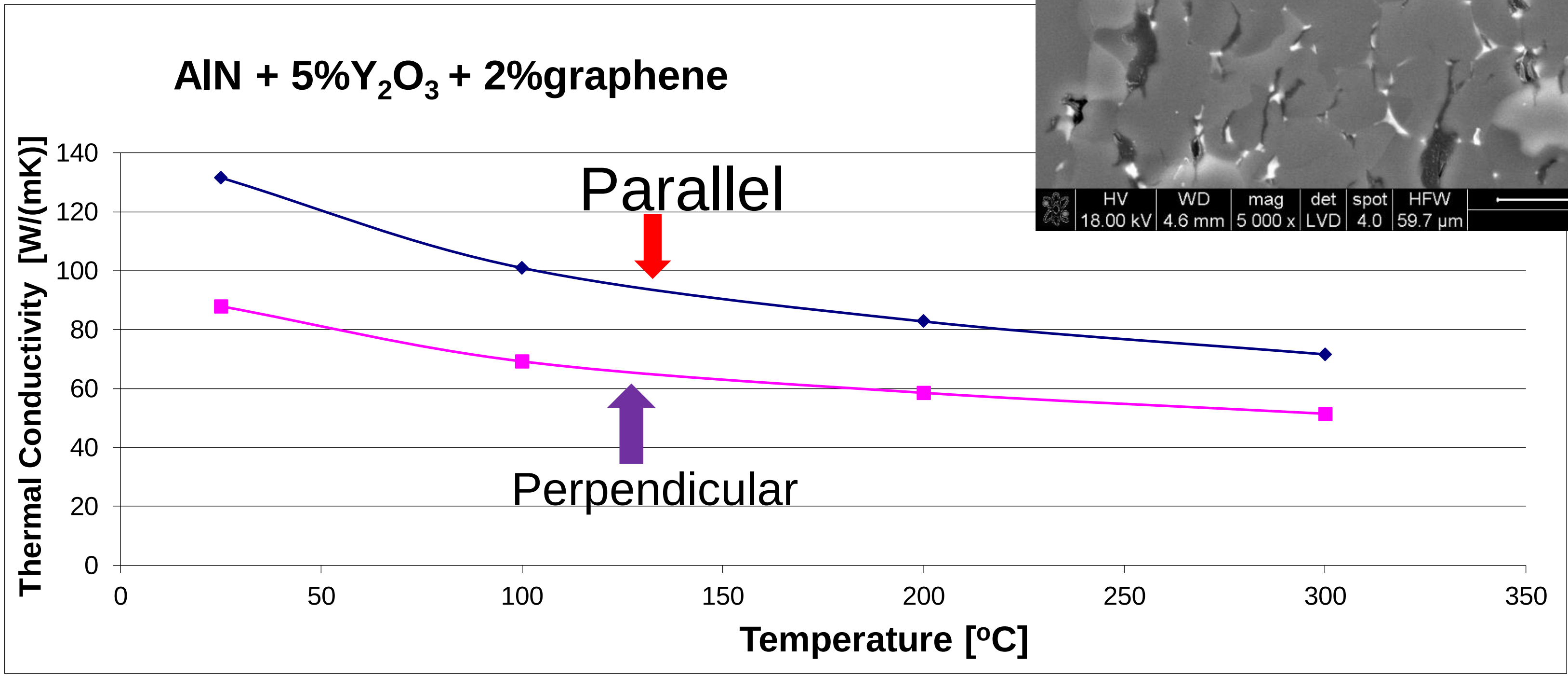
Material	The maximum equivalent diameter d_{2max} [µm]	The average equivalent diameter d_{2mean} [µm]	The average diameter D [µm]
AlN	10.90	4.49	5.88
AlN + 2 wt% GPLs	11.74	5.03	6.53
AlN + 4 wt% GPLs	6.00	2.30	3.01
AlN + 6 wt% GPLs	5.97	2.07	2.66
AlN + 8 wt% GPLs	6.55	2.37	2.94
AlN + 10 wt% GPLs	6.88	2.38	2.96

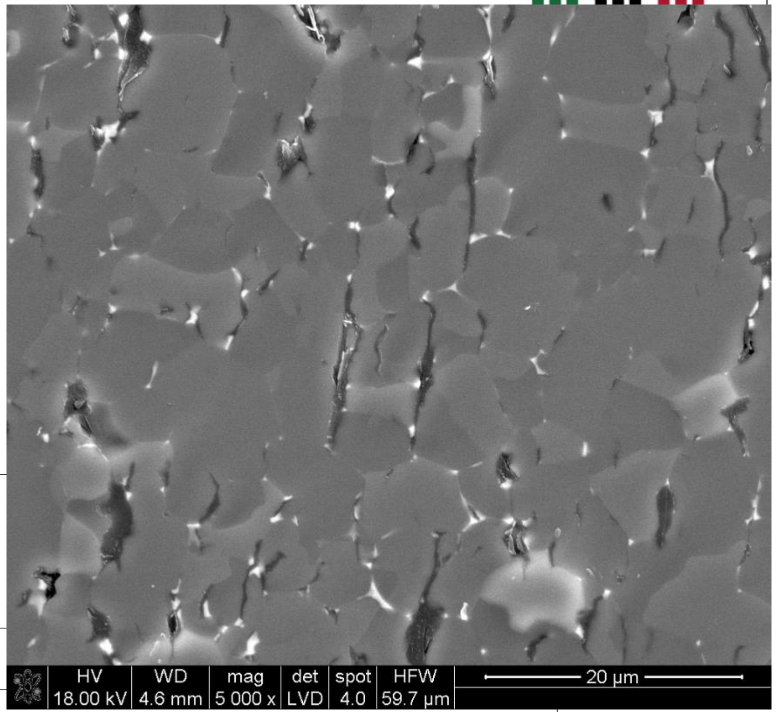


Orientation coefficient

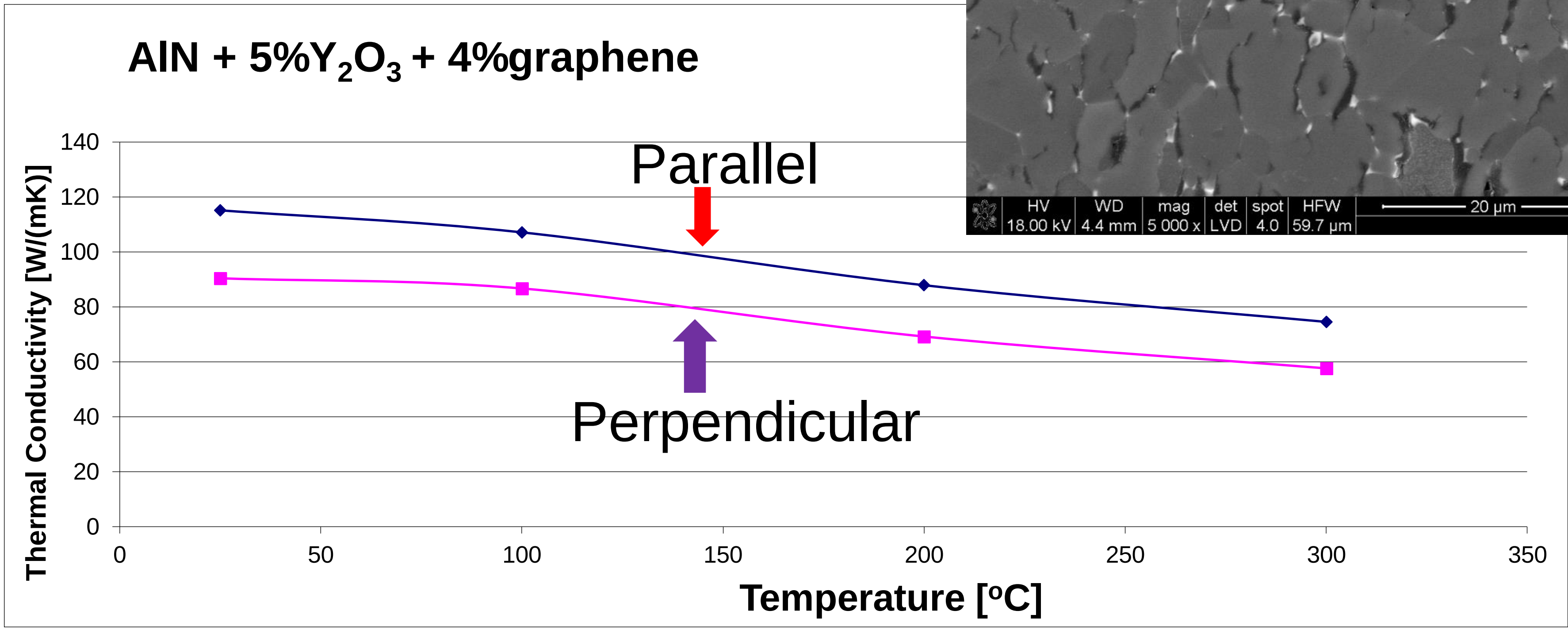


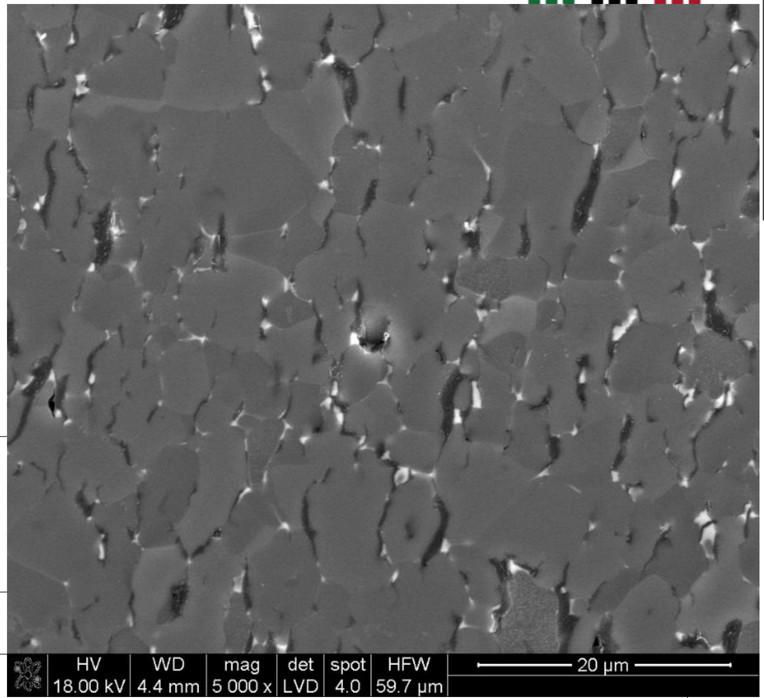
Thermal conductive anisotropy of AlN-graphen nanocomposites



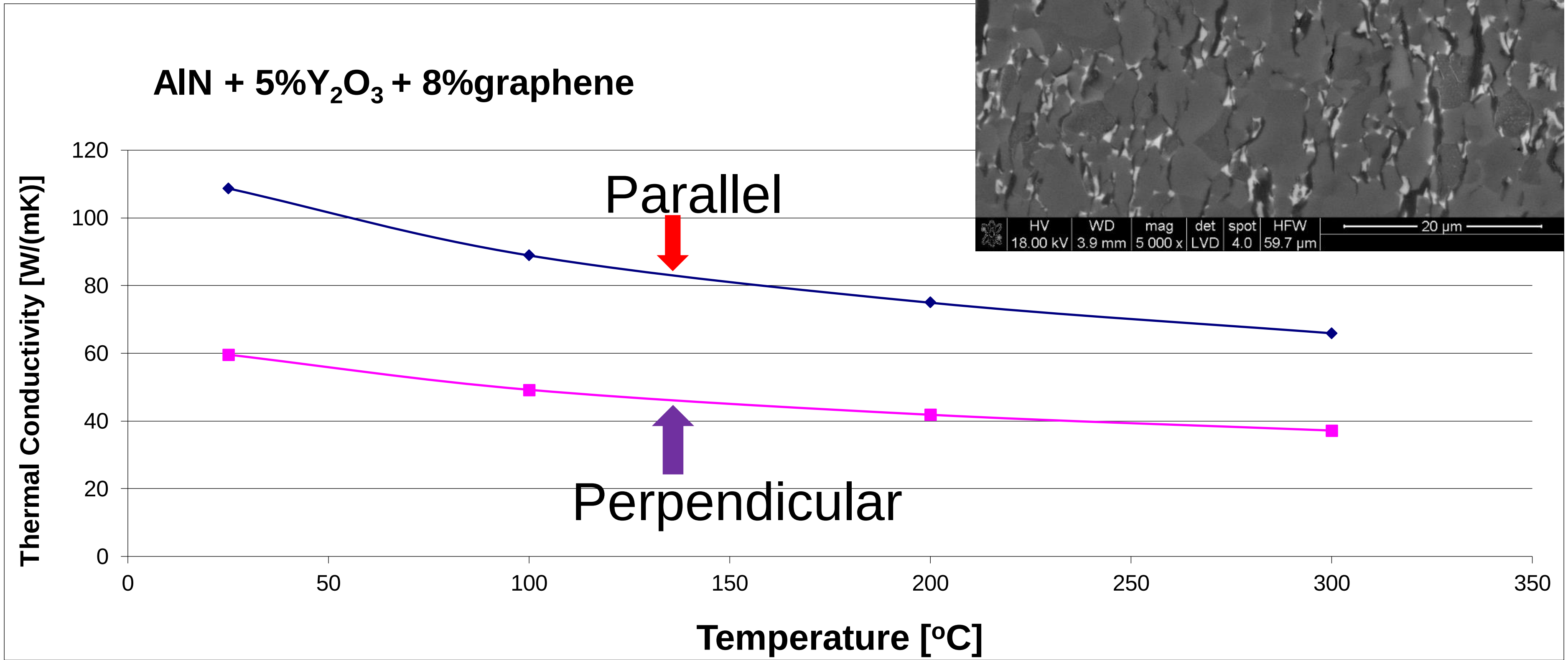


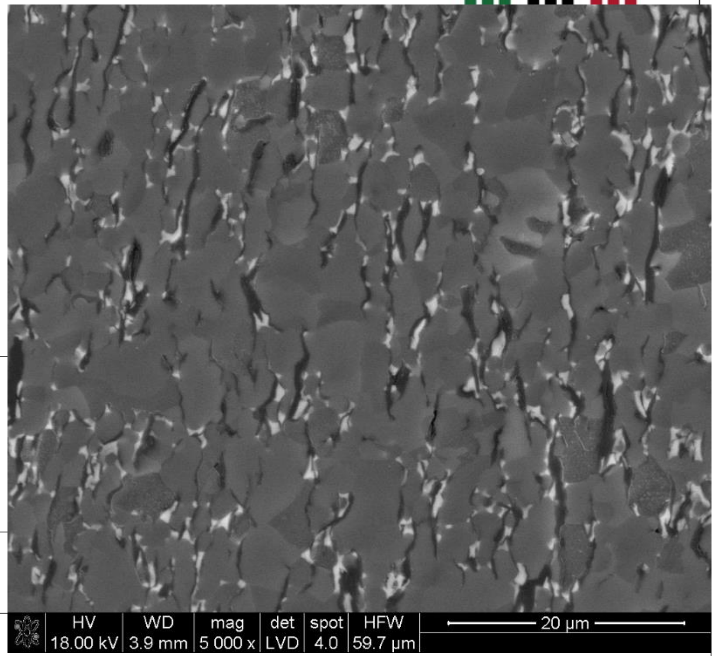
Thermal conductive anisotropy of AlN-graphen nanocomposites



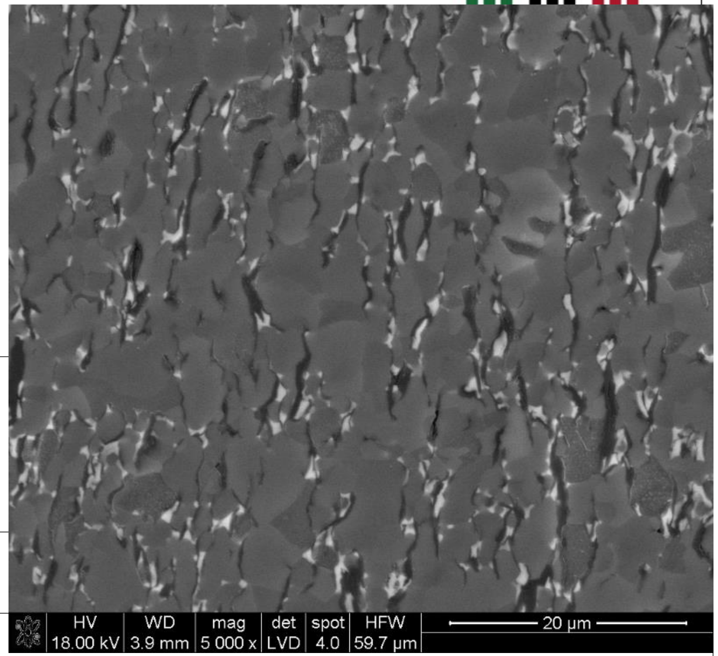


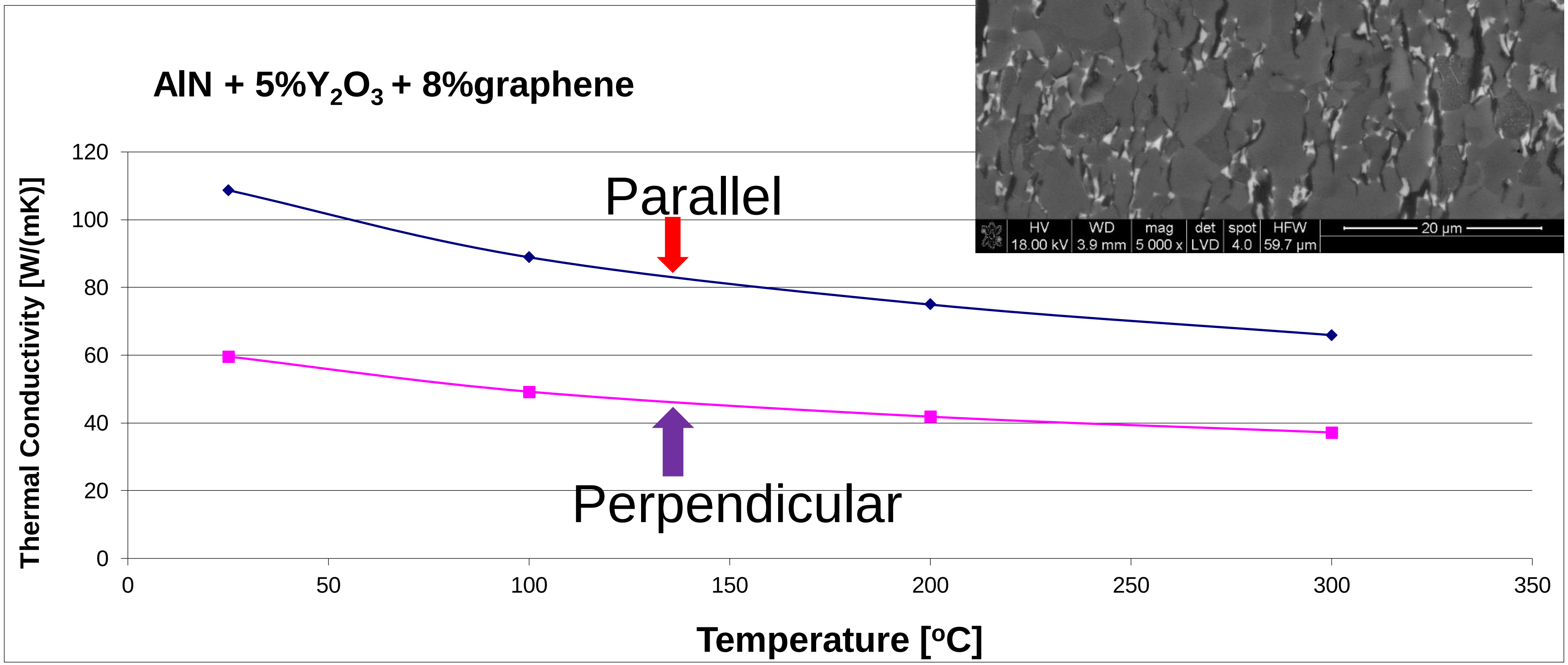
Thermal conductive anisotropy of AlN-graphen nanocomposites





Thermal conductive anisotropy of AlN-graphen nanocomposites

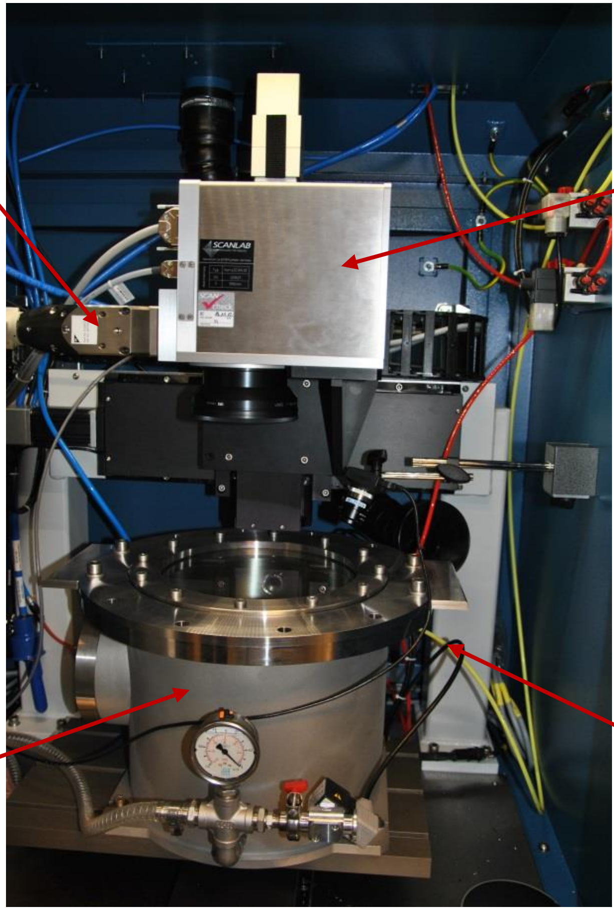




The influence of graphite/graphene additions on Fiber Laser Scanner Processing

Laser beam supply

Two kinds of fibre laser supply: 200 (10 µm accuracy) and 2000kW (50 µm accuracy)

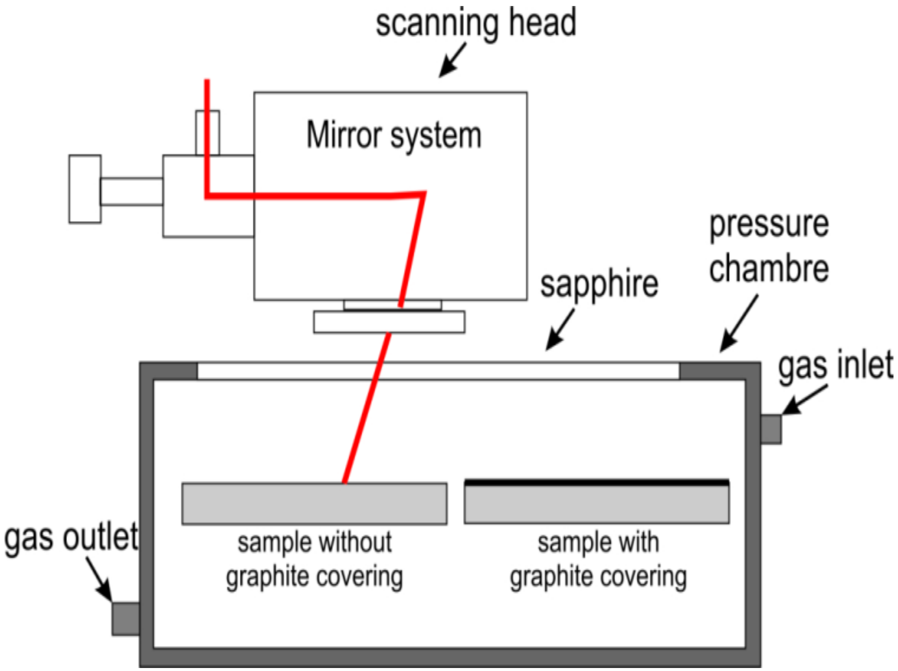


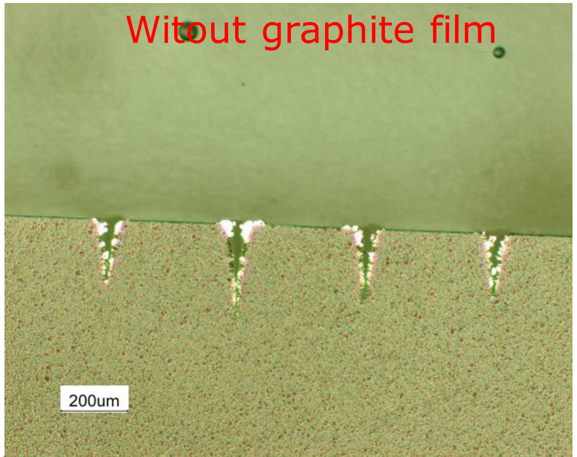
Scanner head

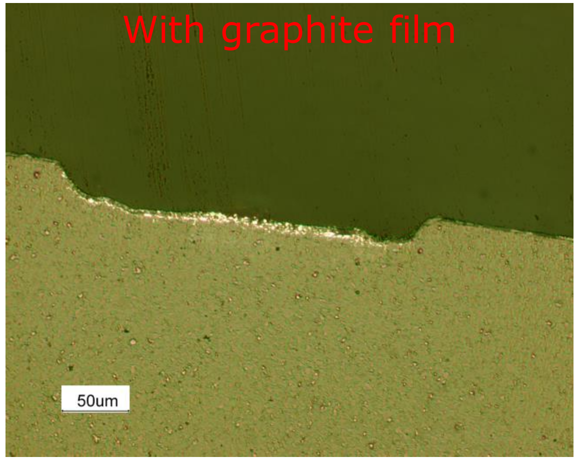
Specially designed pressure chambre with possibility of temperature control

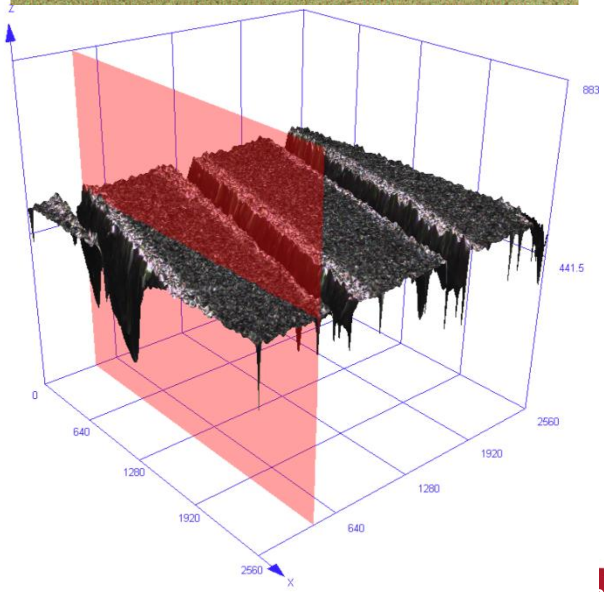
Nitroger gas 5 bars pressure

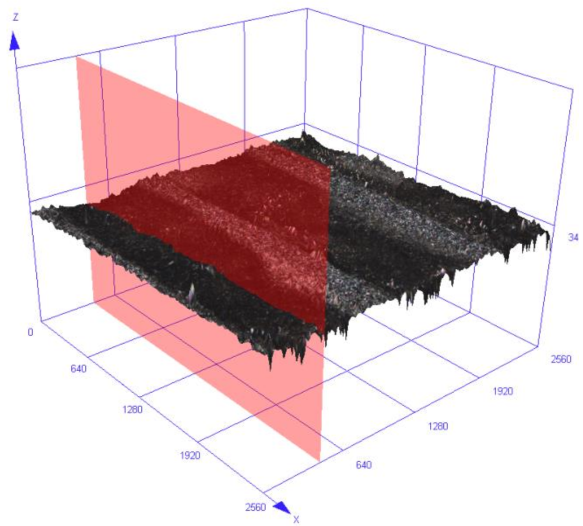
Fiber Laser Scanner AlN processing

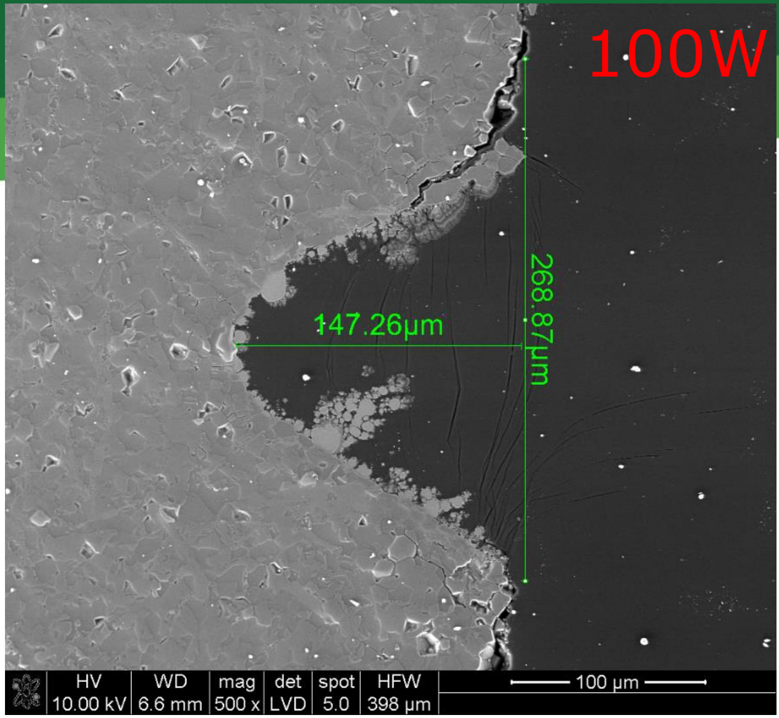


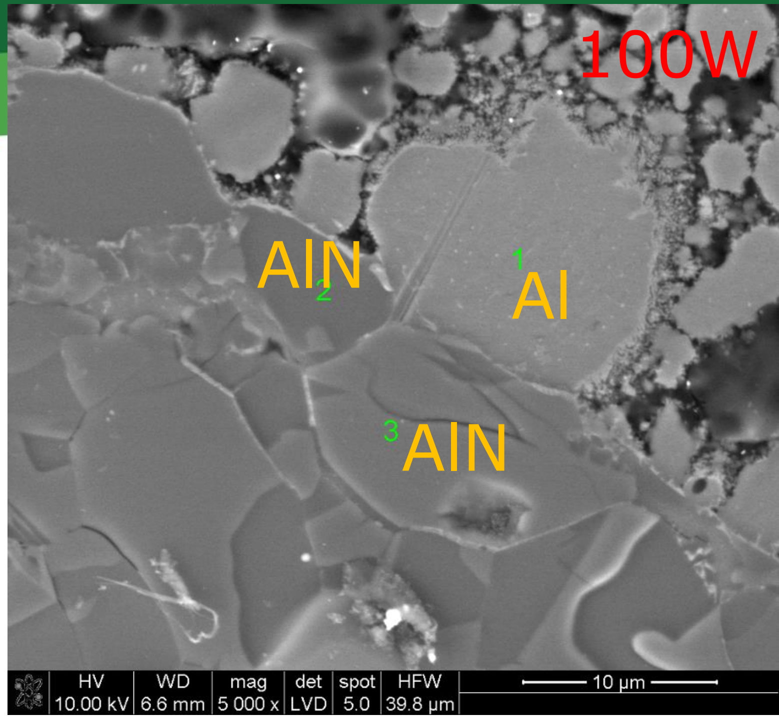












AGH

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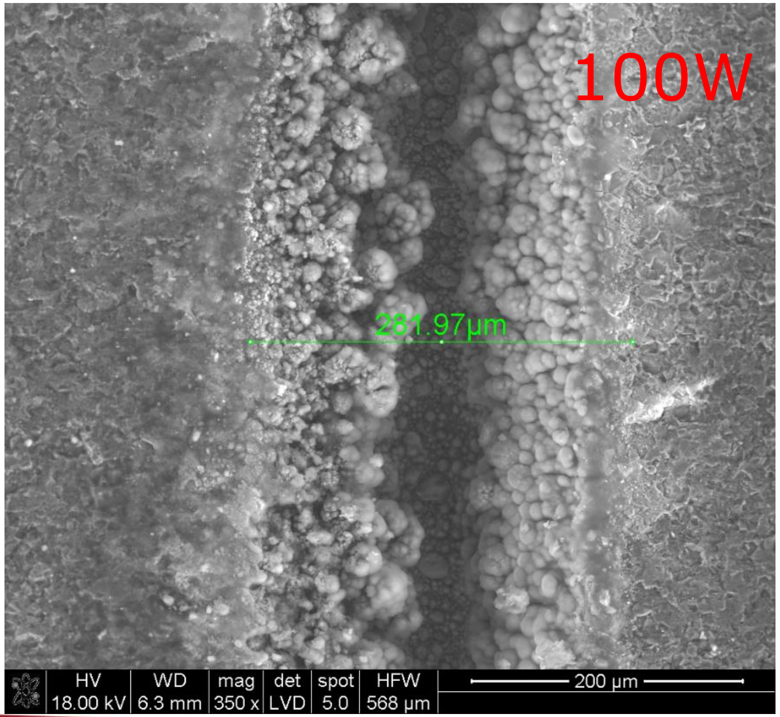
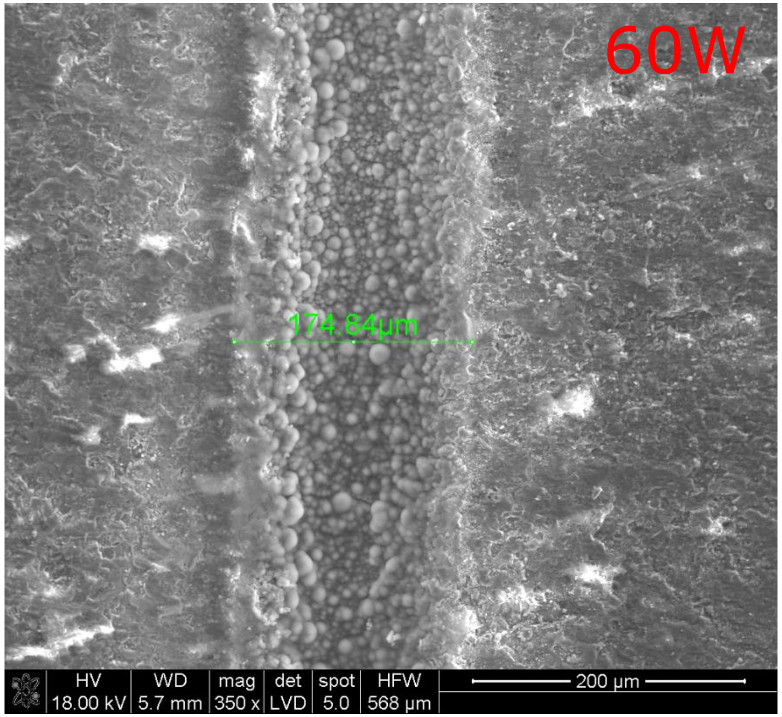
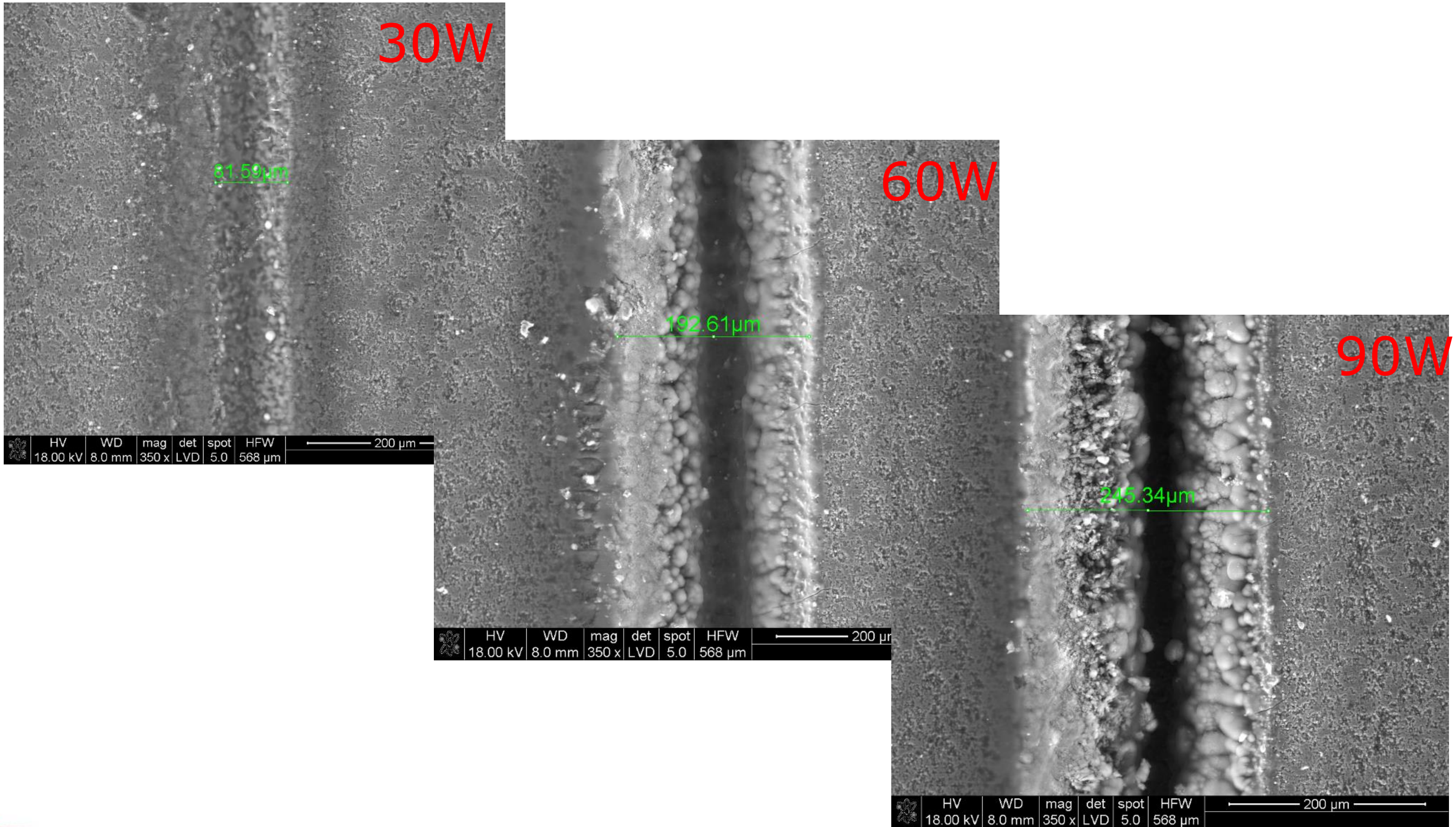
Ministerstwo Nauki i Szkolnictwa Wyższego



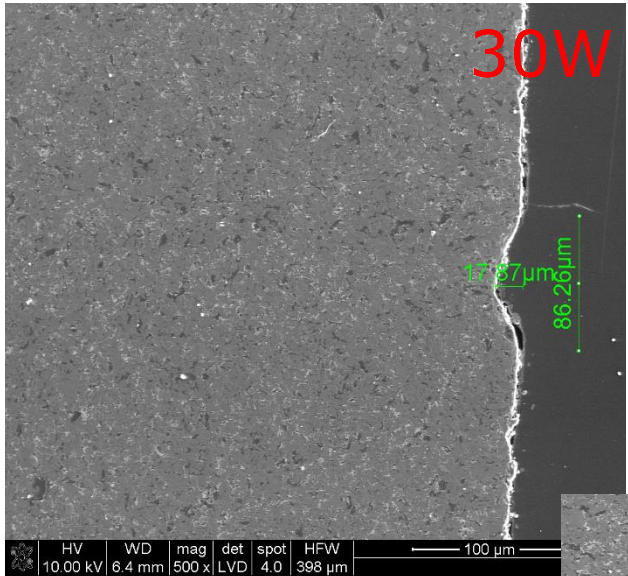


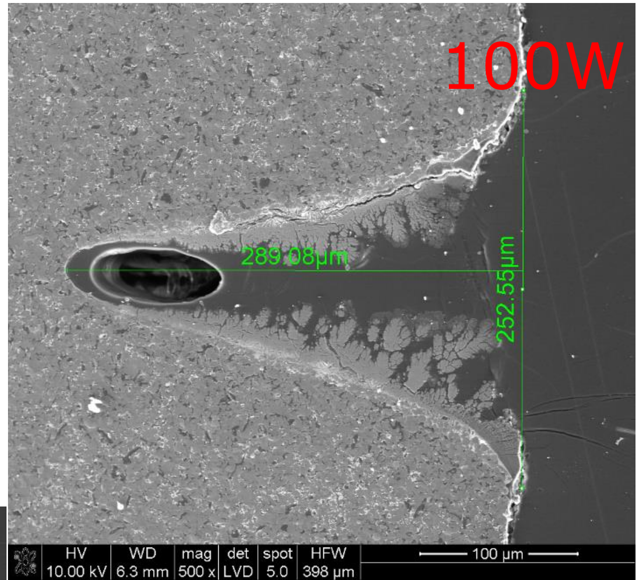
AlN laser shpaing

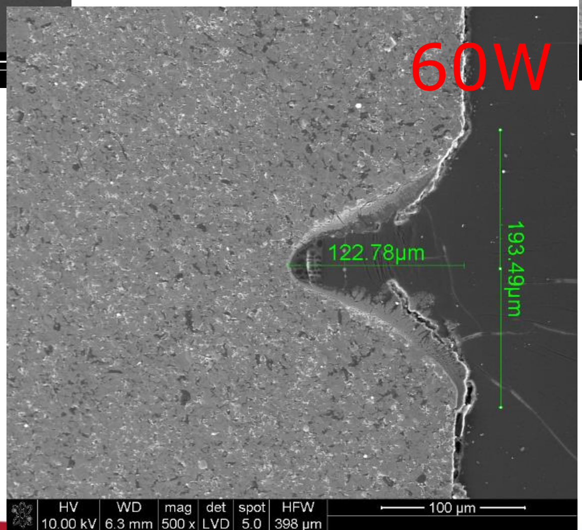
Laser processing of AlN + 10 wt% GPLs composites



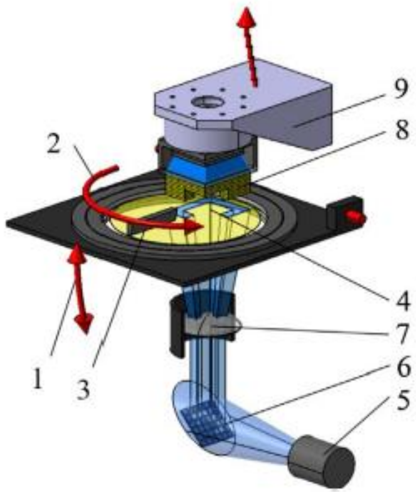
Laser processing of AlN + 10 wt% GPLs composites







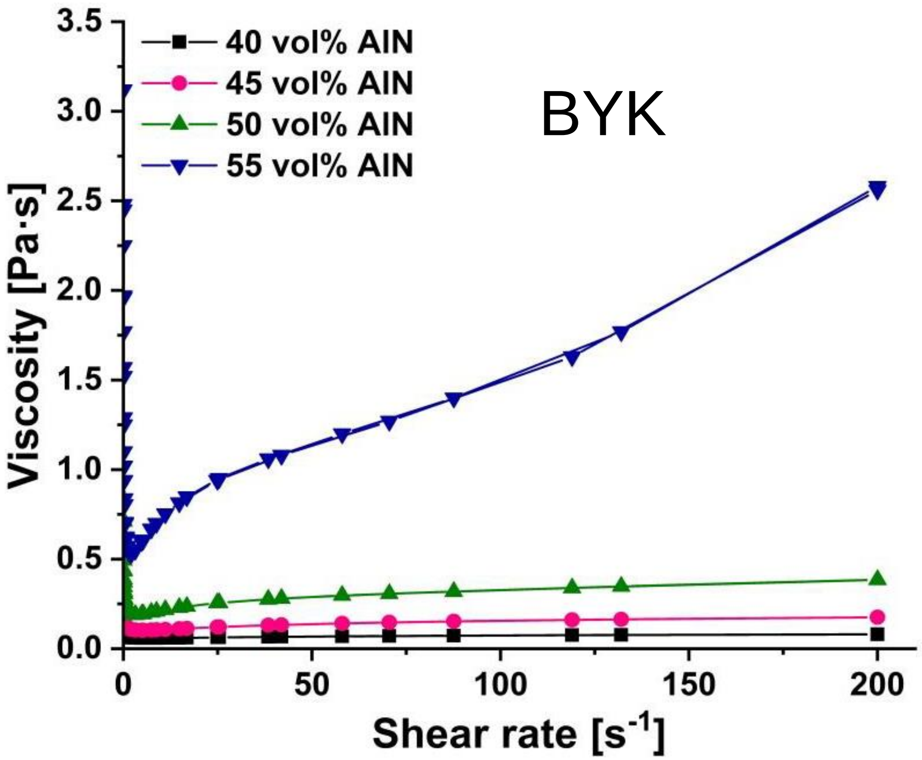
Additive manufacturing of AlN by UV-resin AlN suspension

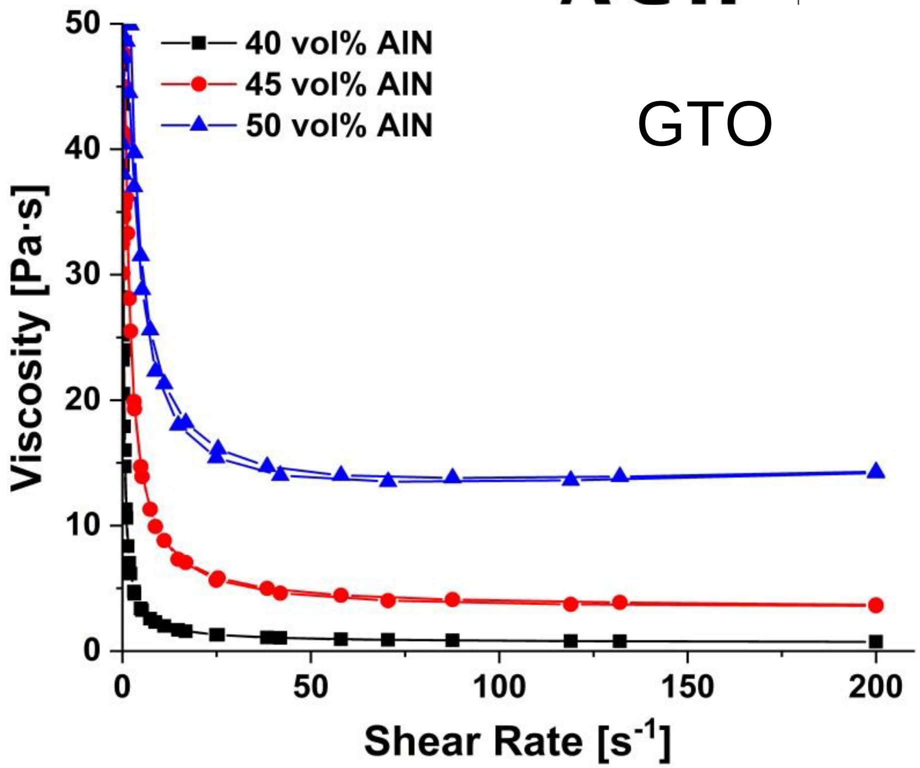


1. Tilting mechanism,
2. Rotating mechanism,
3. Coating blade,
4. Exposed slurry,
5. LED light source,
6. DMD chip,
7. Optics,
8. Green part,
9. Z-stage

CeraFab 7500 3D printer from Lithoz (Austria)

Influence of different dispersing agent on rheological behaviour of AlN slurries

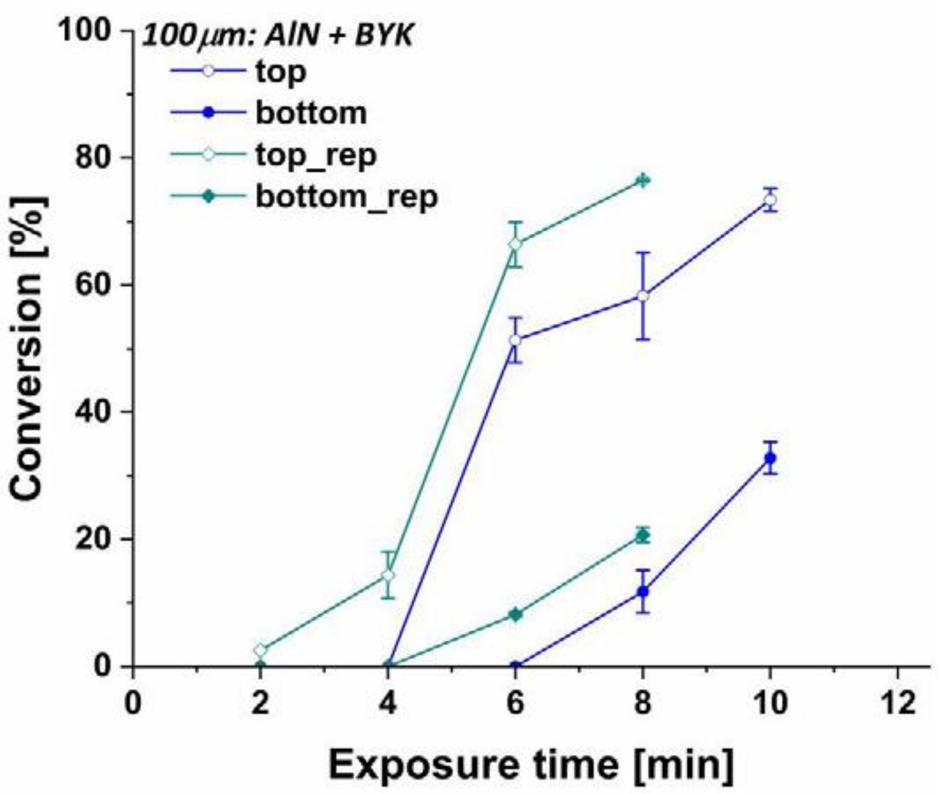


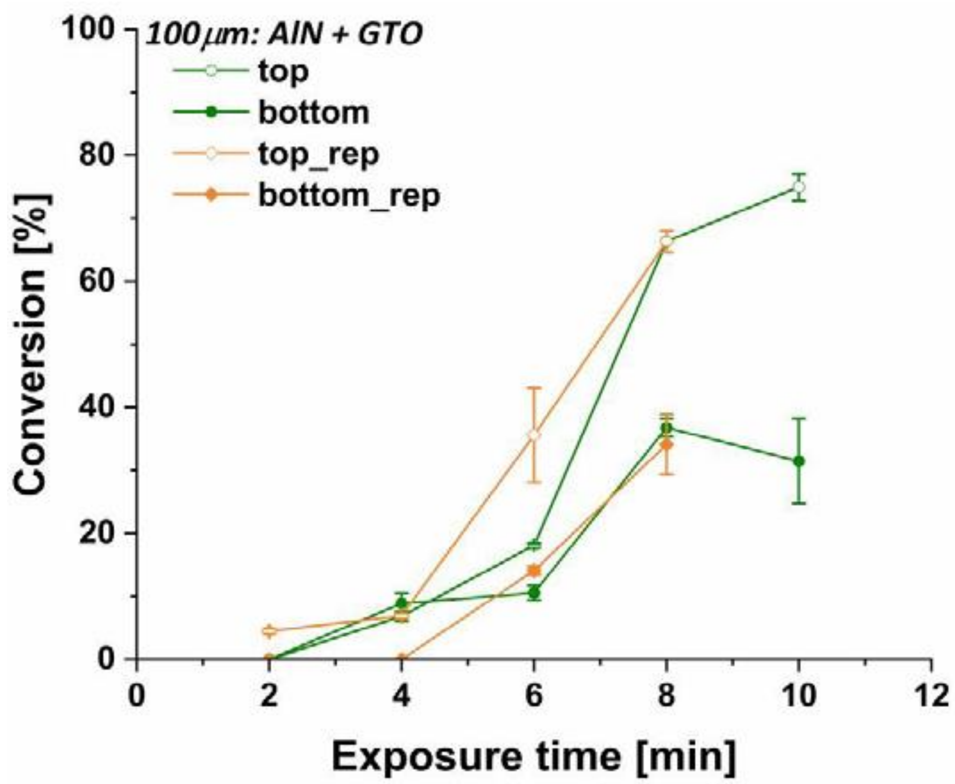


Two different dispersing agents were examined; BYK-W 9010 from Additives & Instruments (Germany) and glyceryl trioleate (GTO) from Sigma Aldrich (Switzerland)

P. Ożóg, D. Kata, T. Graule, Tape casting of UV-curable aluminium nitride-based slurries, Ceram. Int. 44 (2018) 22800–22807. doi:10.1016/j.ceramint.2018.09.071.

Conversion degree of AlN slurries influenced by exposure time





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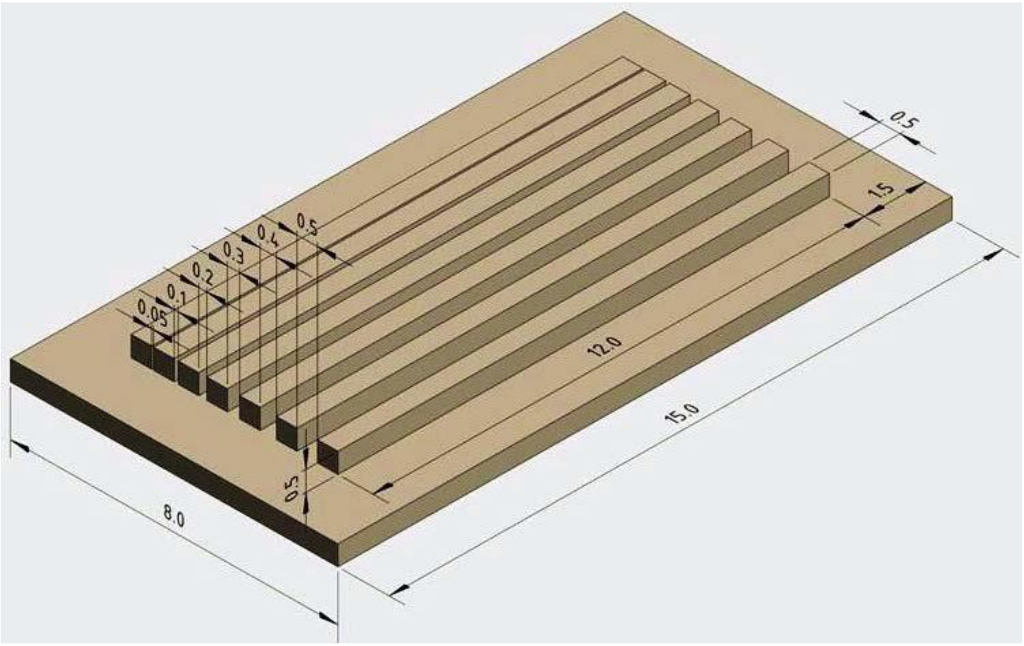
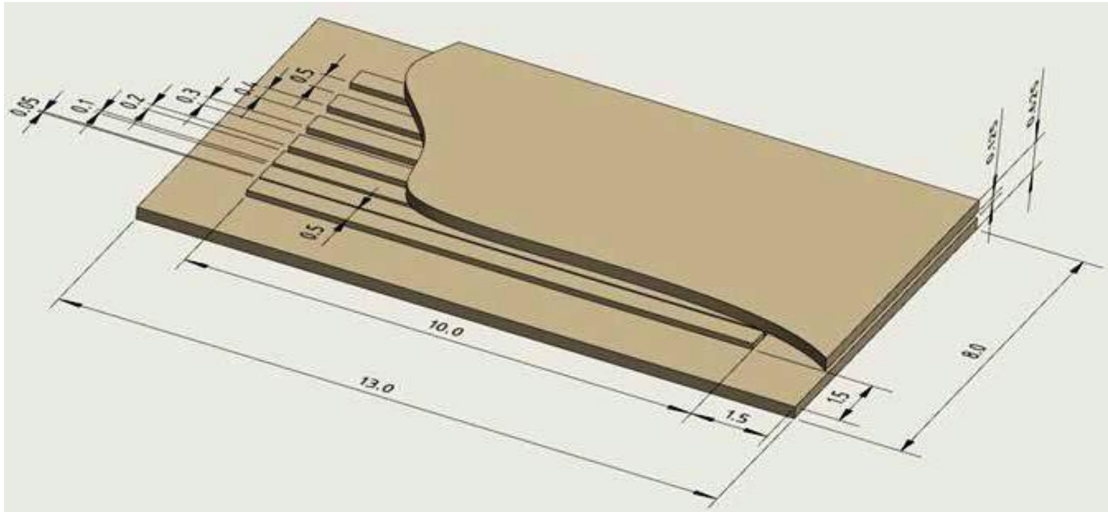
Microheat exchanger test designs

a)

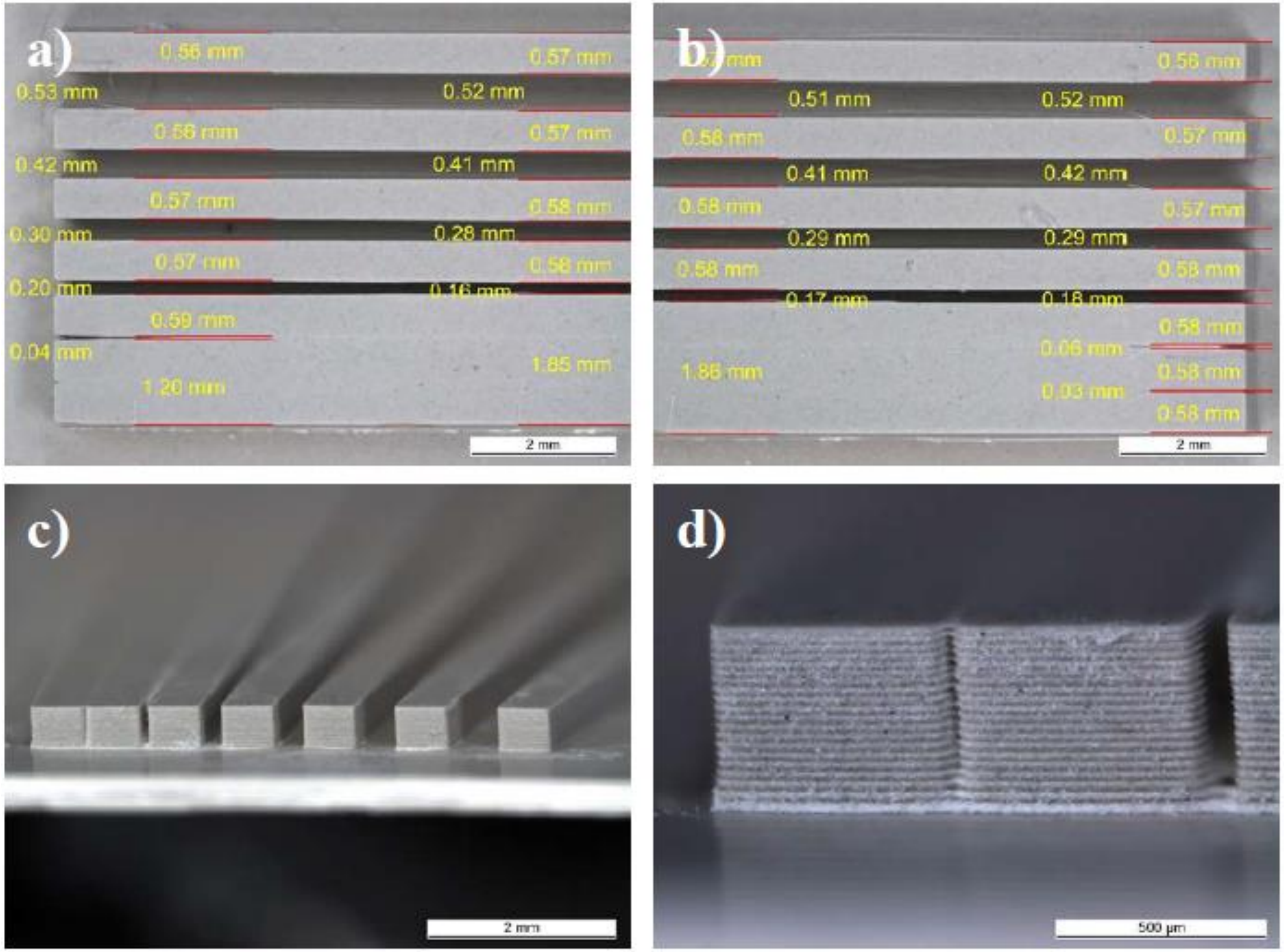


b)

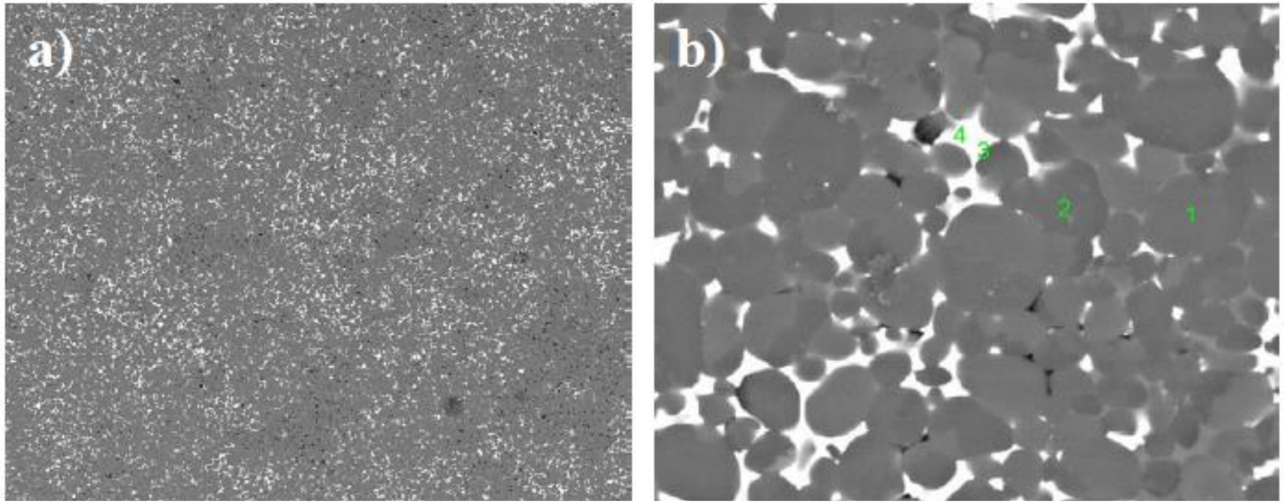


Microheat exchanger test designs: a) plate with channels (PCH);
b) plate with channels and cover (PCHC) [drawing prepared by Roland Bätchold, Empa]

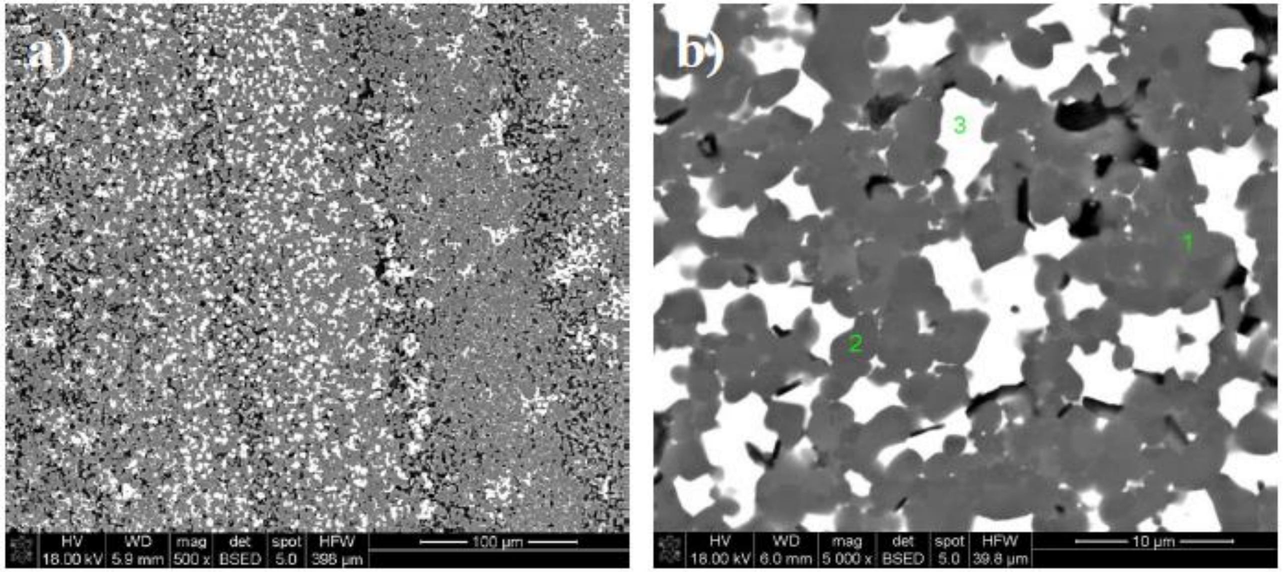
AlN microheat exchanger additive manufactured



AlN microstructure examination



AlN microstructure prepared by conventional presurelless sintering



AlN microstructure prepared by additive manufacturing

Rapid high-energy ceramics processes (RHEP)

Using of high-energy sources for local synthesis or consolidation of powders (economic processing)

↓

Rapid local heating of materials
(SHS, Laser Manufacturing, Laser Sintering, Spark Plasma Sintering etc.)

↓

Rapid temperature growth and, after the process, very rapid cooling

↓

Rapid physicochemical phenomena during reaction, sintering etc.

↓

Possibility to prepared unique compounds

↓

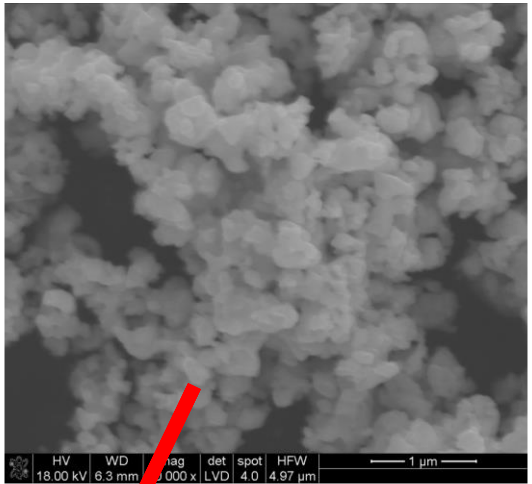
Examples:

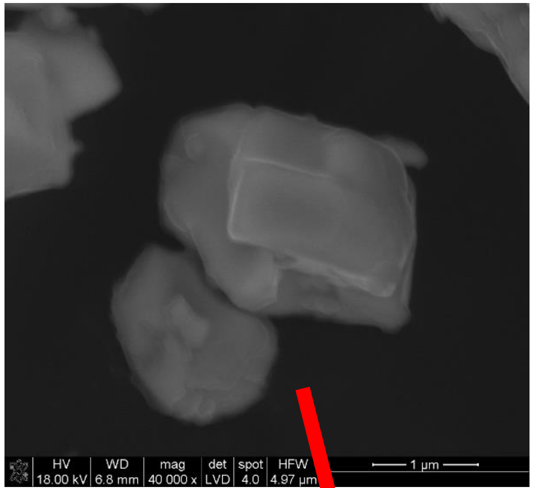
1. Self-Propagating High-Temperature Synthesis (SHS),

2. Laser Manufacturing (LM)

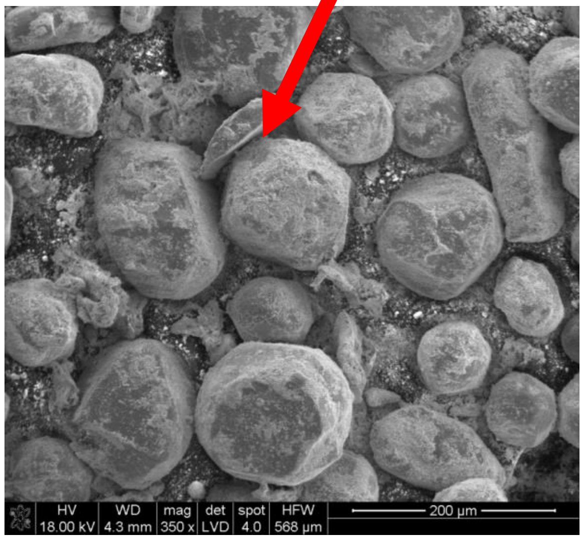
Inconel 625 – WC particles mixture preparation

WC ≈ 0.64 µm



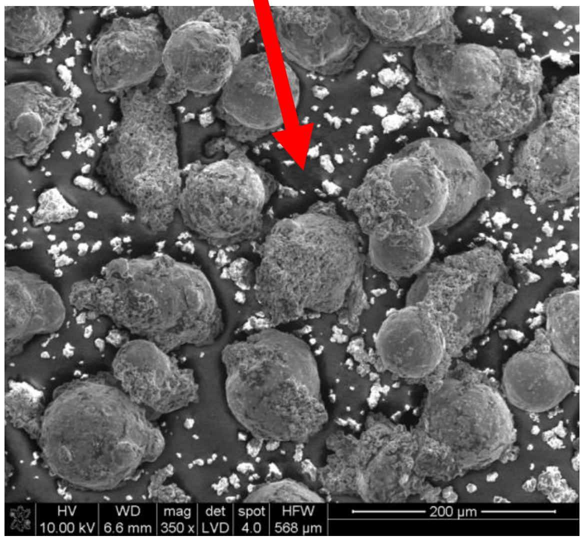


WC ≈ 6.03 µm



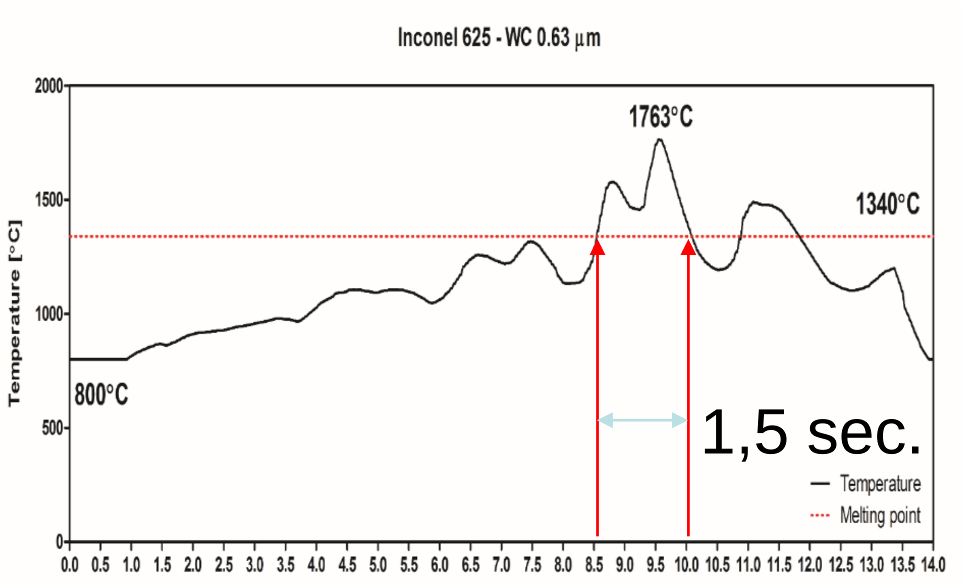
Inconel 625 + WC (0.64 µm) mixture

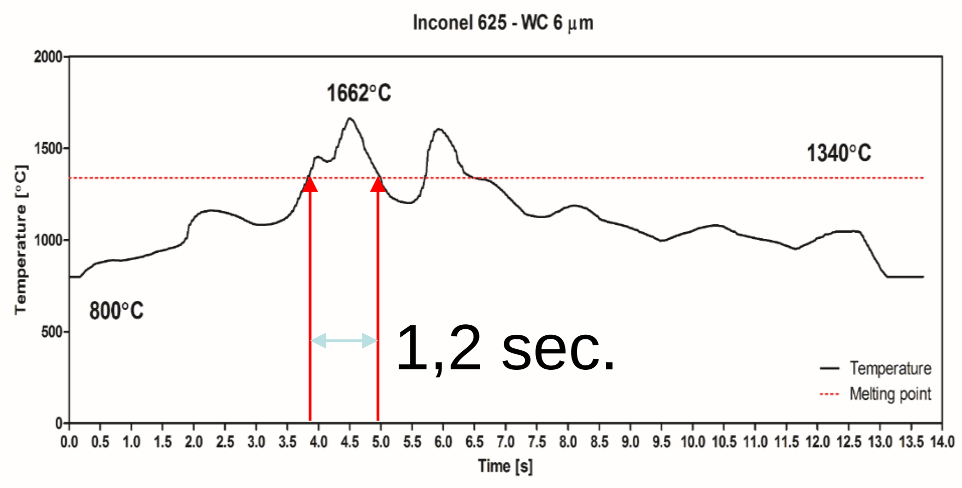
wet homogenization for 2 hours including 0,5% resin



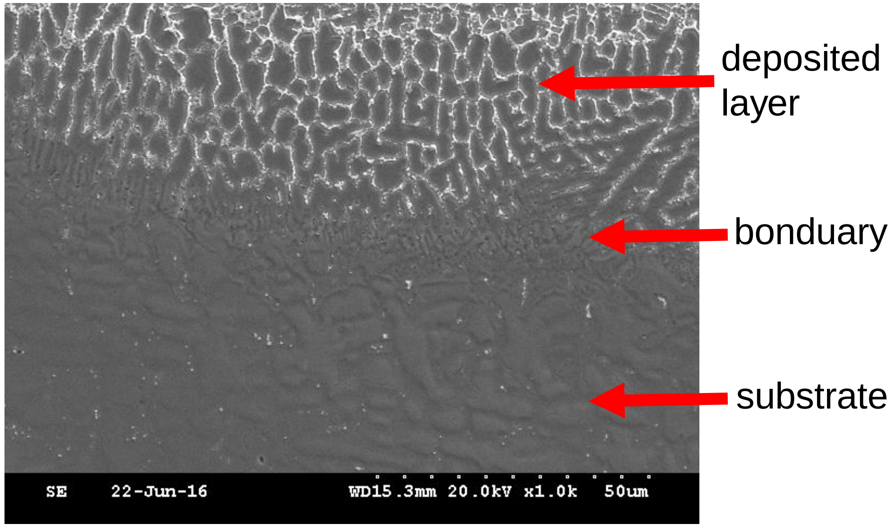
Inconel 625 + WC (6.03 µm) mixture

3D manufacturing of Inconel – WC composites

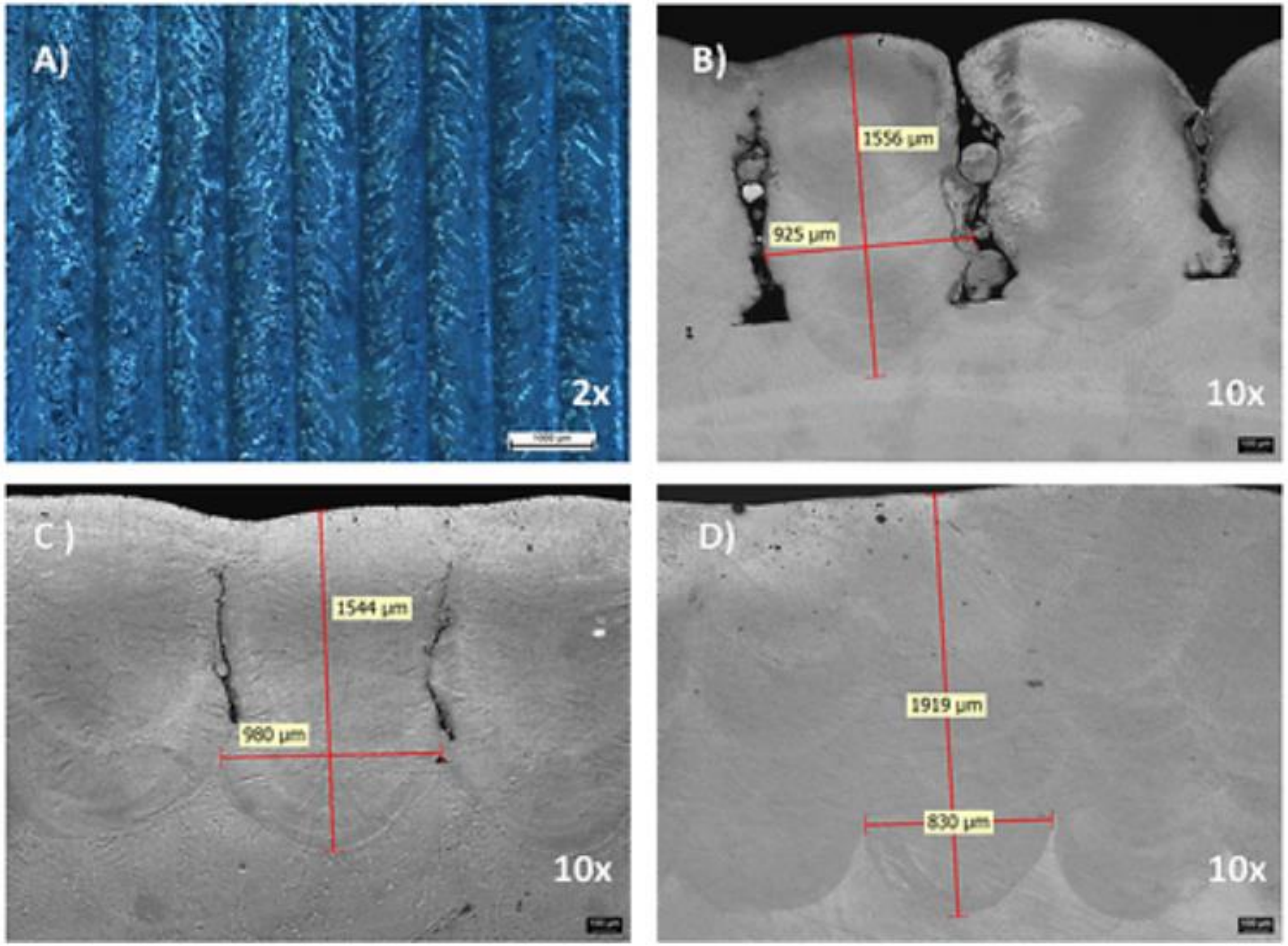




Parameter	1st attempt	2nd attempt
Track length	10 mm	10 mm
Amount of consecutive tracks	10	10
Amount of sublayers	6	6
Laser beam diameter	500 µm	500 µm
Distance between center of tracks	1 mm	0.8 mm
Nominal laser power	220 W	320 W
Scanning velocity	10 mm/s	10 mm/s

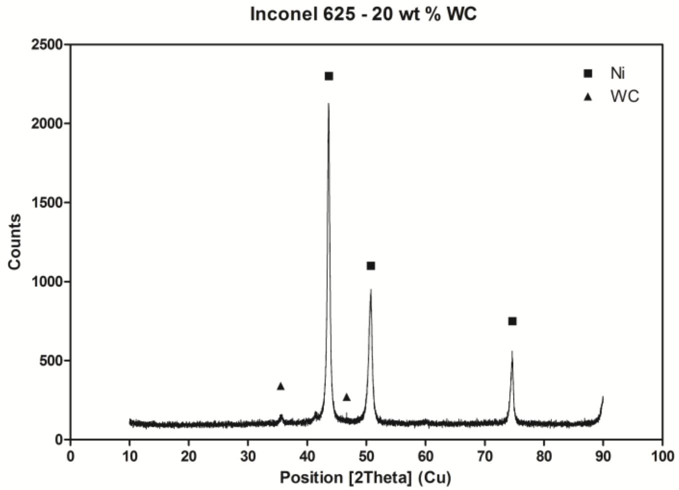


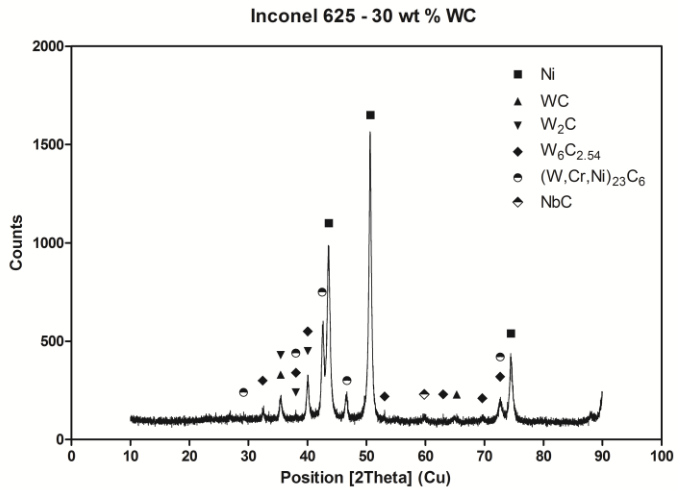
3D manufacturing of Inconel – WC composites

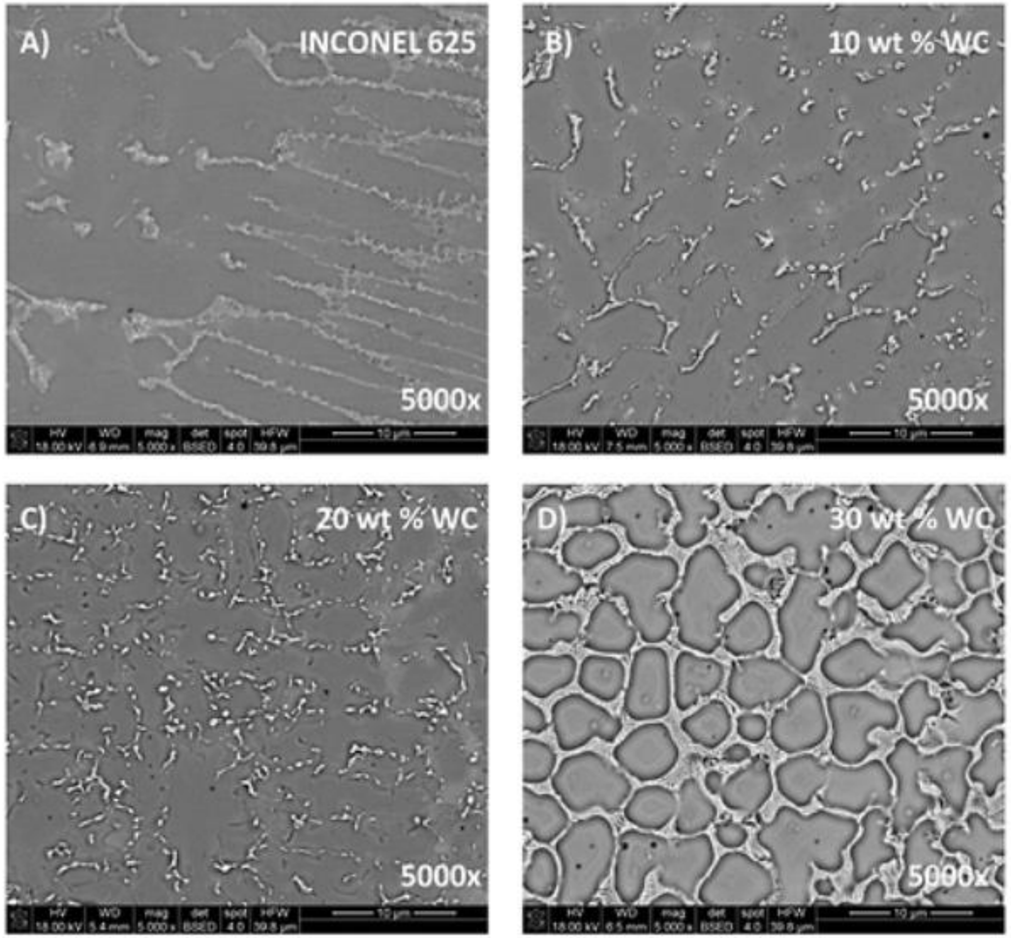


Microstructure appearance of laser shpaed layers

XRD and microstructure analysis of Inconel 625 - 10%, 20% and 30% WC additions

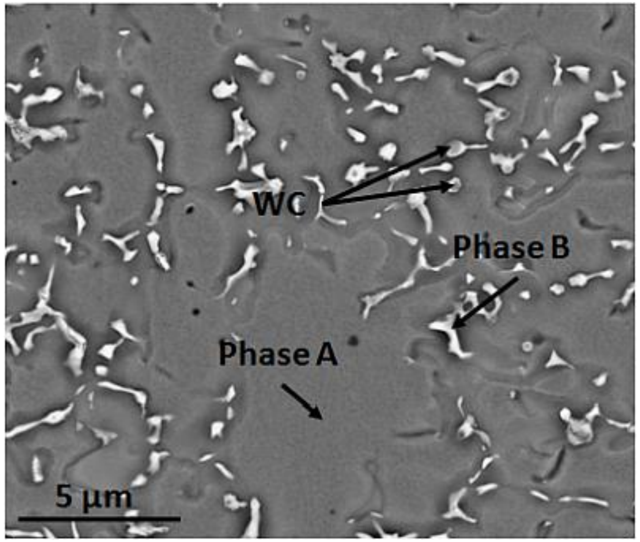


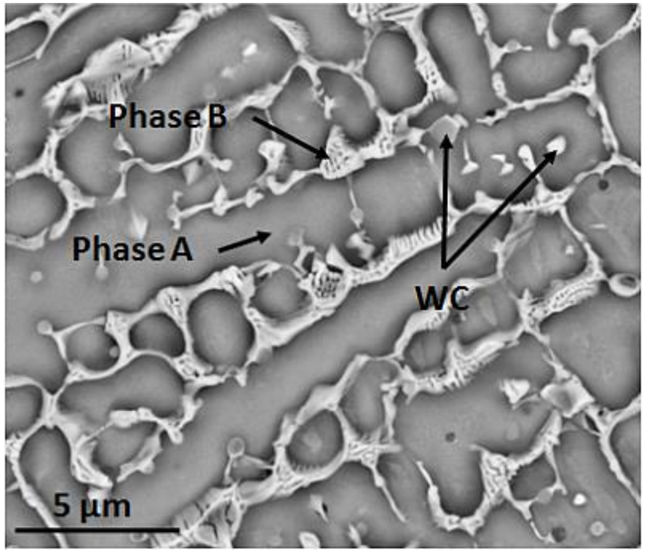




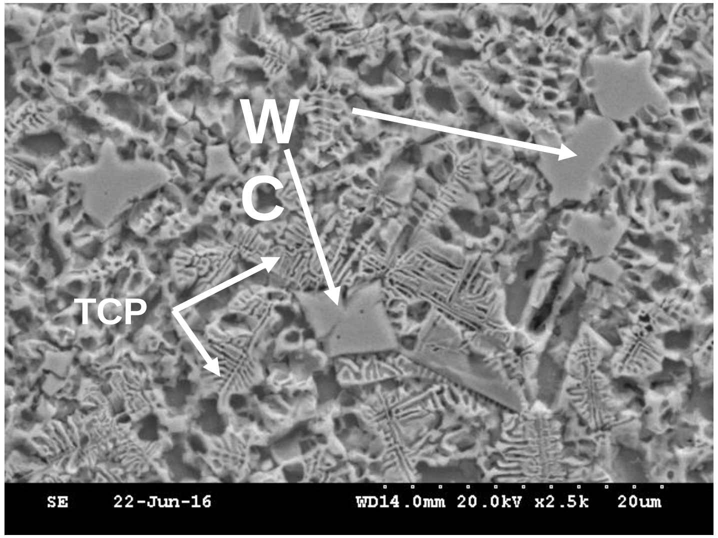
Microstructures of Inconel 625 – WC 0,63µm

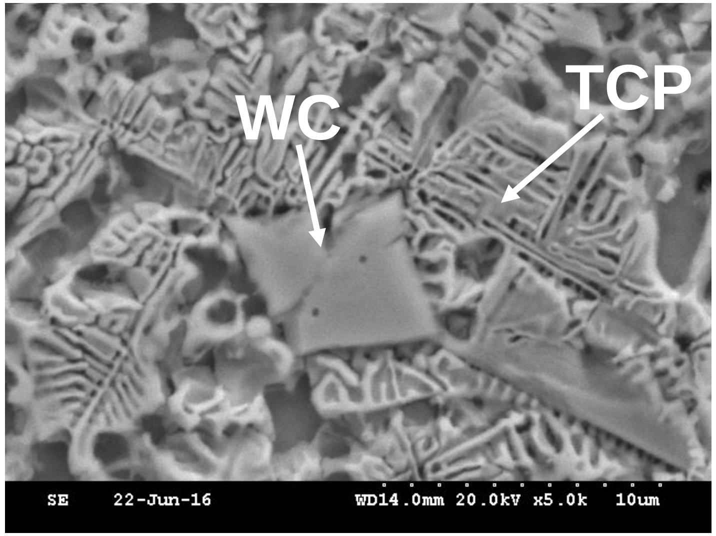
Inconel 625 + WC (0.64 µm) composite





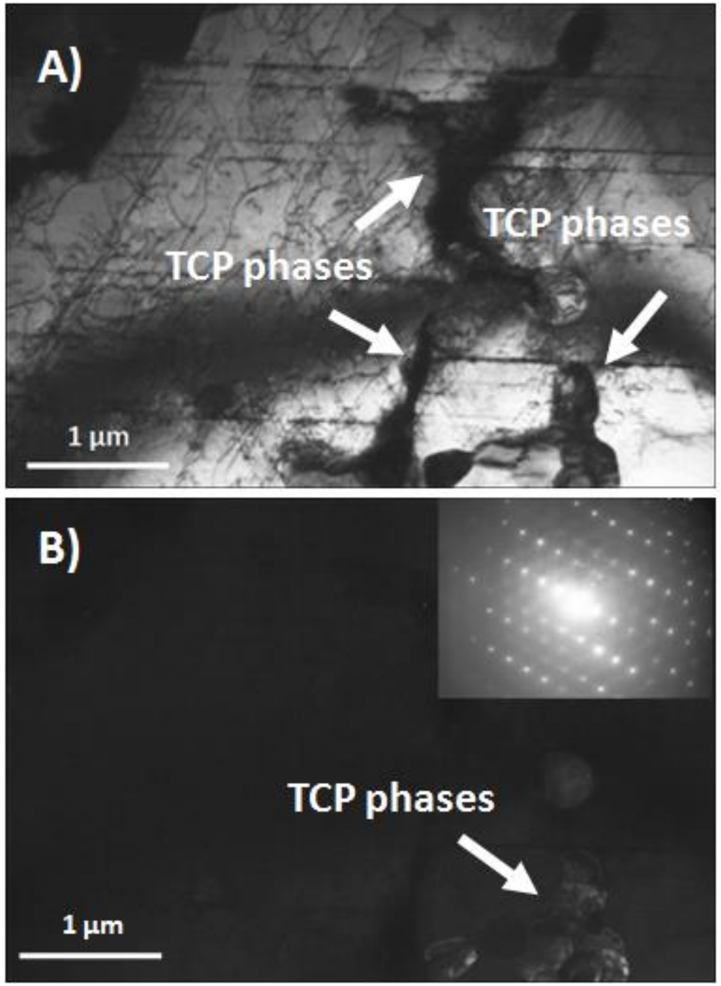
Inconel 625 + WC (6.03 µm) composite



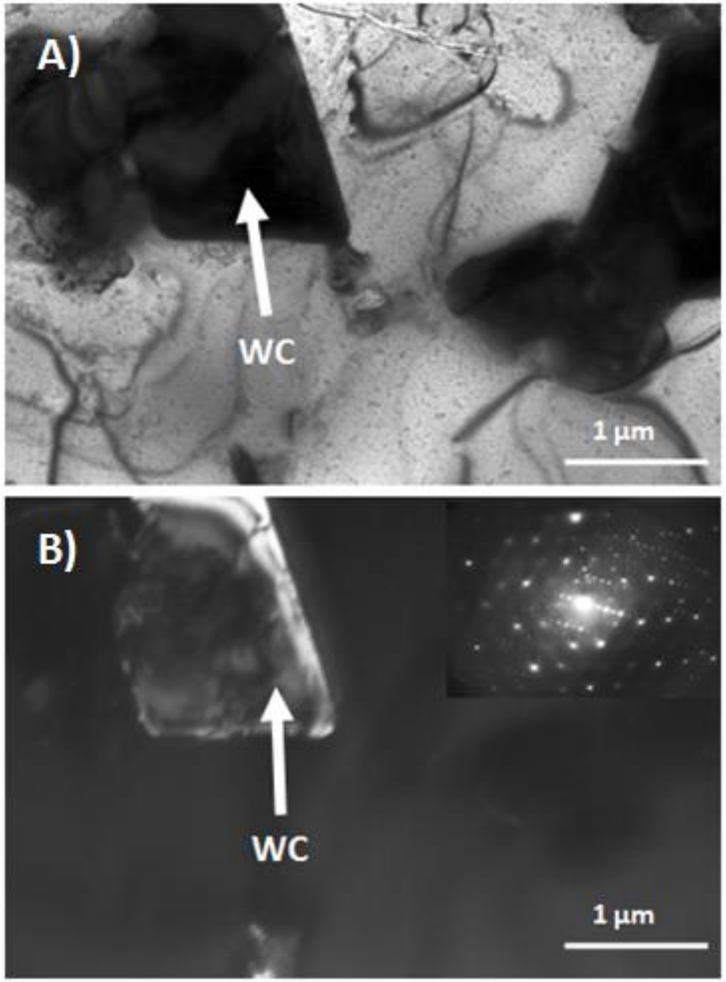


TEM analysis of laser 3D shaped of Inconel – WC composites

Inconel 625 + WC (0.64 μm) composite



Inconel 625 + WC (6.03 μm) composite



3D shaping of silica „hollow fibre”

$$V_{Total} = V_{vdW} + V_{elect} + V_{steric}$$

$$V_{VdW} = -\frac{A_H r}{6\pi x} \qquad x<<r$$

$$A = a\left(\frac{\varepsilon_m - \varepsilon_p}{\varepsilon_m + \varepsilon_p}\right)^2 + b\frac{\left(n^2{}_m - n^2{}_p\right)^2}{\left(n^2{}_m + n^2{}_p\right)^{3/2}}$$

V_{Total-} total energy balance of dispersion

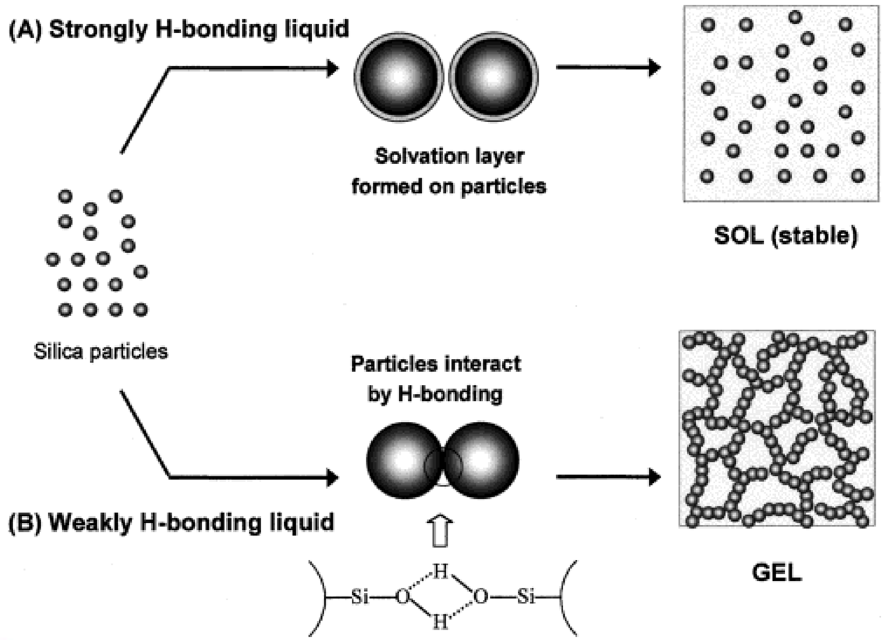
x : distance

r : particle radius

A_H: effective Hamaker constant

ε-dielectric constant (m-monomer,p-particle)

n-refractive index (m-monomer p-particle)

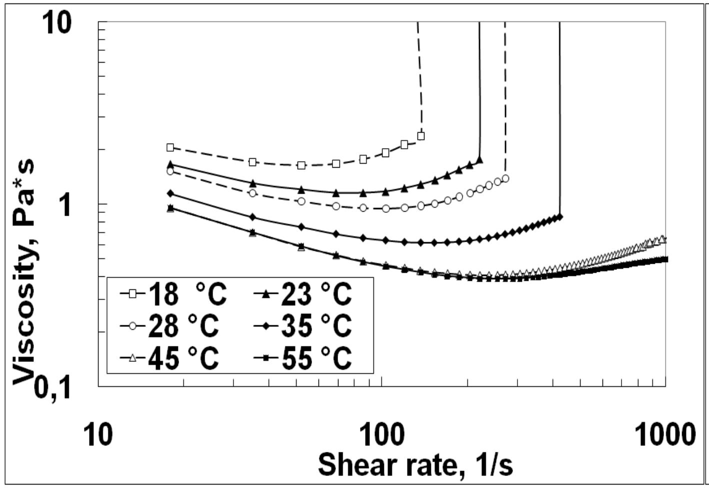


Hydrophylic monomers (monoacrylates with –OH grups)
high maximum concentration of powder, shear thickinning behavior after critical shear rate

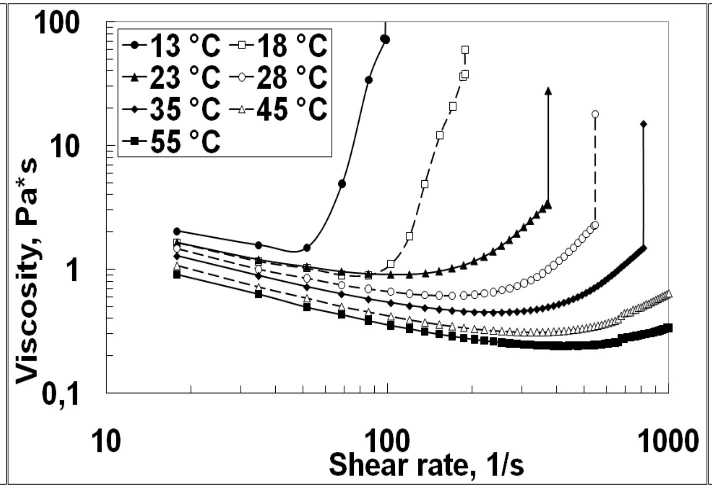
Hydrophobic monomers (diacrylates)-
very low maximum solid loading (shear thinning behavior at these concentration)

Raghavan, S. R.,Walls, H. J. and Khan, S. A., Rheology of silica dispersions in organic liquids: new evidence for solvation forces dictated by hydrogen bonding. *Langmuir*, 2000, **16**, 7920–7930

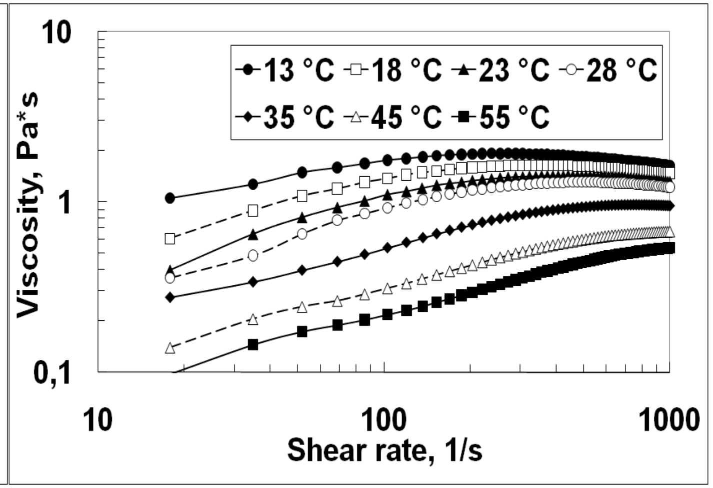
Rheological behaviour of silica dyspersion for 3D shaping



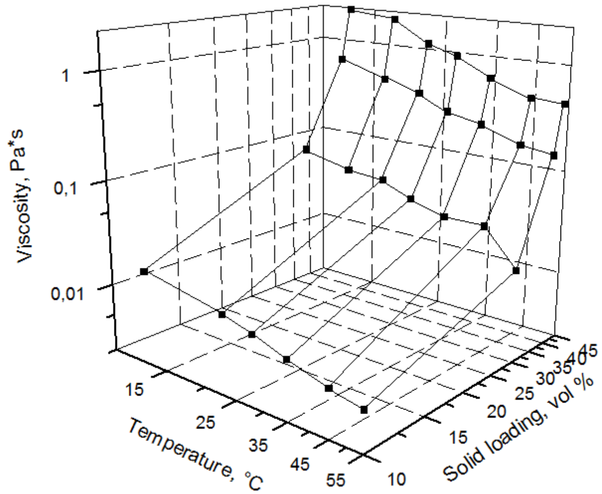
43 % obj. nanokrzemionki w PEG200DA / 2-HEA



54 % obj. krzemionki 0,25 mikrometra w PEG200DA /2-HEA

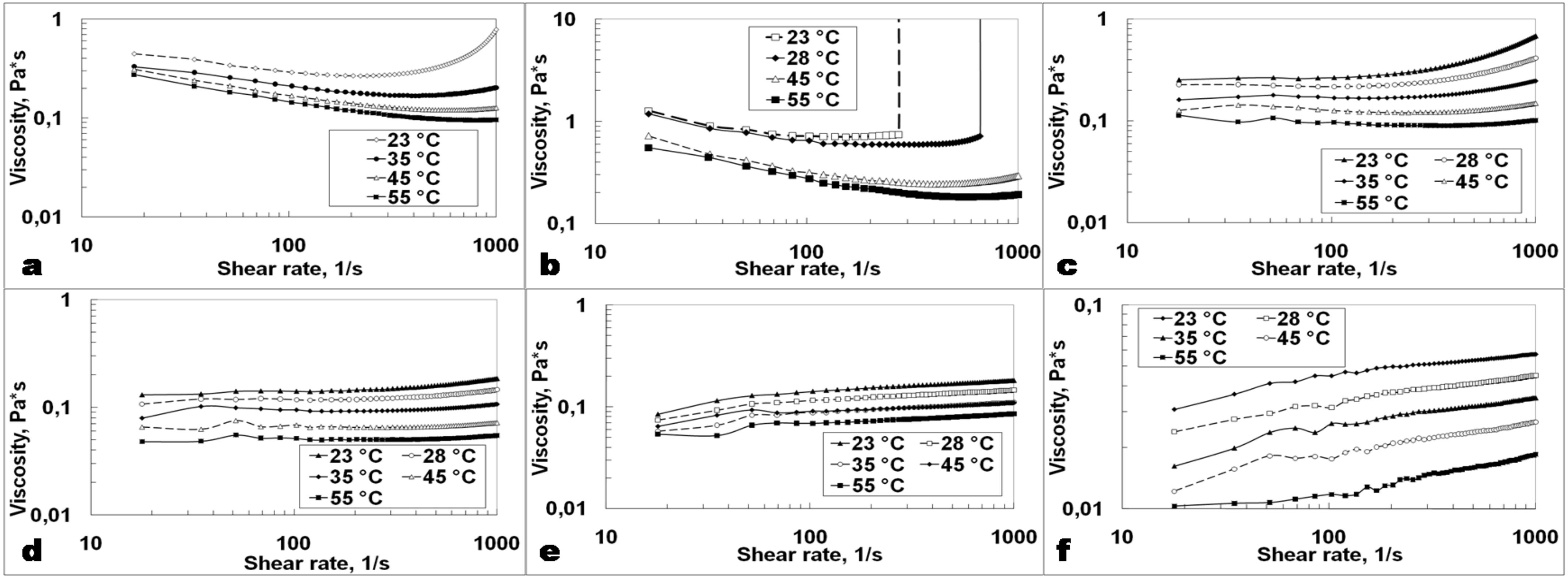


60 % obj. mikrokrzemionki w PEG200DA / 4-HBA



Temperature influence on viscosity of silica dyspersions

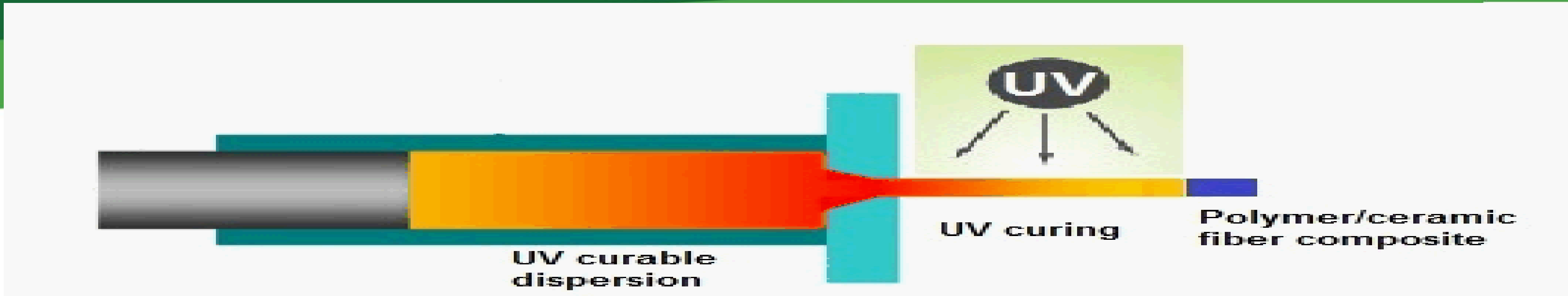
Rheological behaviour of silica dyspersion for 3D shaping

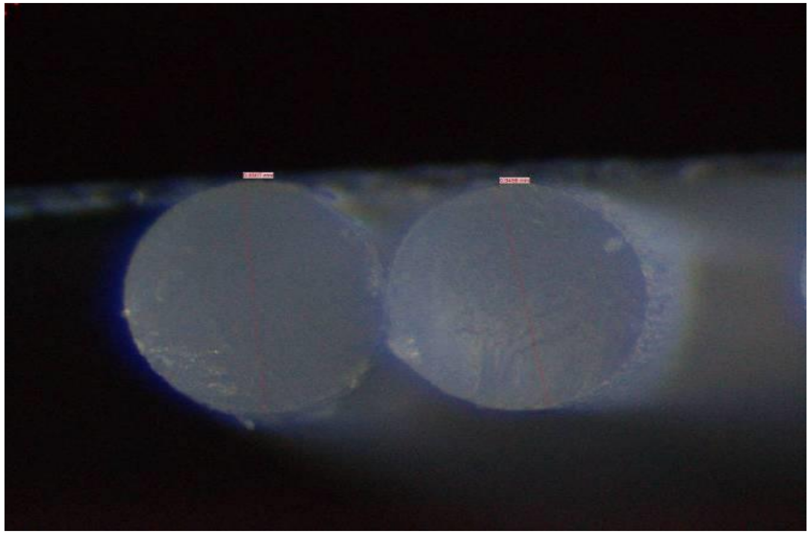


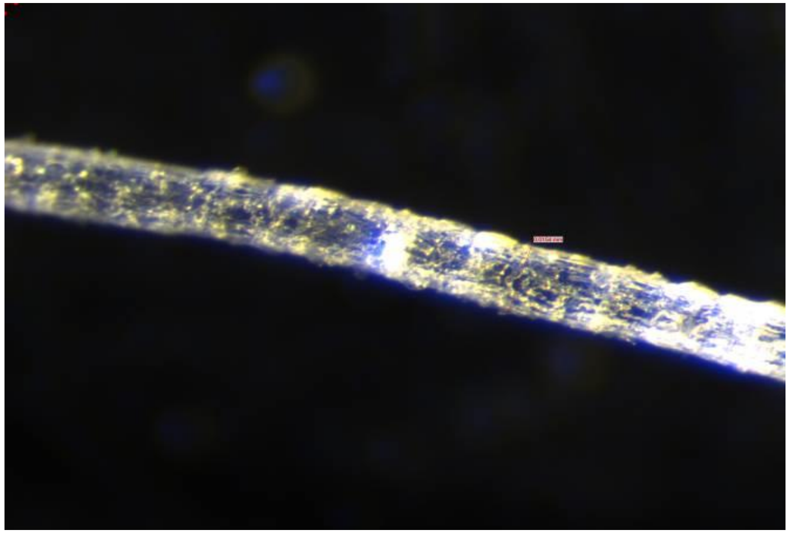
a) 40 % obj. “nano” + 5 % obj.“mikron”, b) 40 % obj.“nano” + 10 % obj.“mikron”, c) 30 % obj.“nano” + 20 % obj.“mikron”,

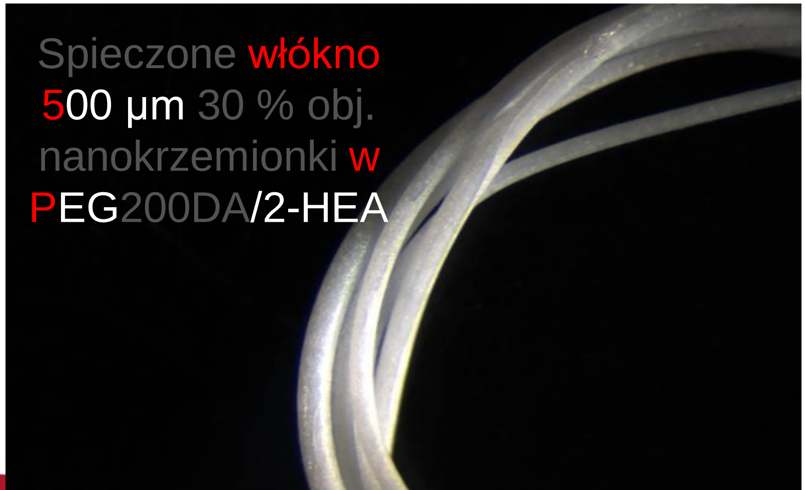
d) 25 % obj.“nano” + 25 % obj. “mikron”, e) 15 % obj.“nano” + 35 % obj.“mikron”, f) 10 % obj.“nano” + 35 % obj.“mikron”,

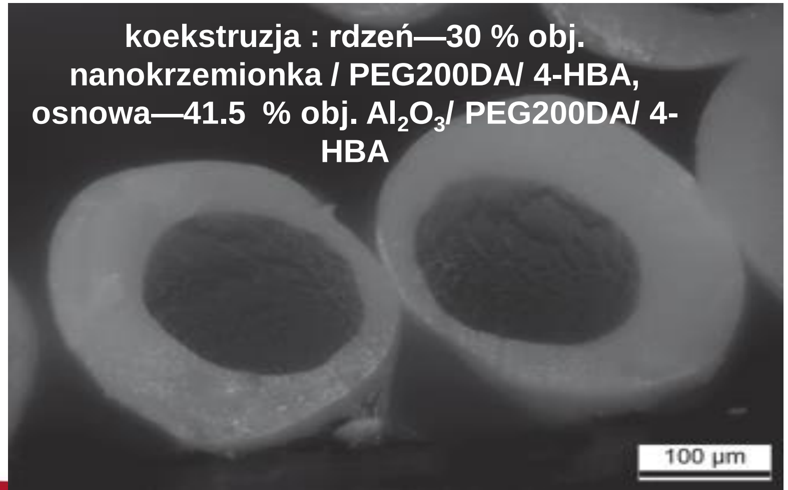
Maciej Woźniak, Dariusz Kata, Thomas Graule, UV Curable silica Disperions for Rapid Prototyping Applications, J. European Ceramic Soc. 2012


UV
UV curable dispersion
UV curing
Polymer/ceramic fiber composite






Spieczone włókno 500 µm 30 % obj. nanokrzemionki w PEG200DA/2-HEA

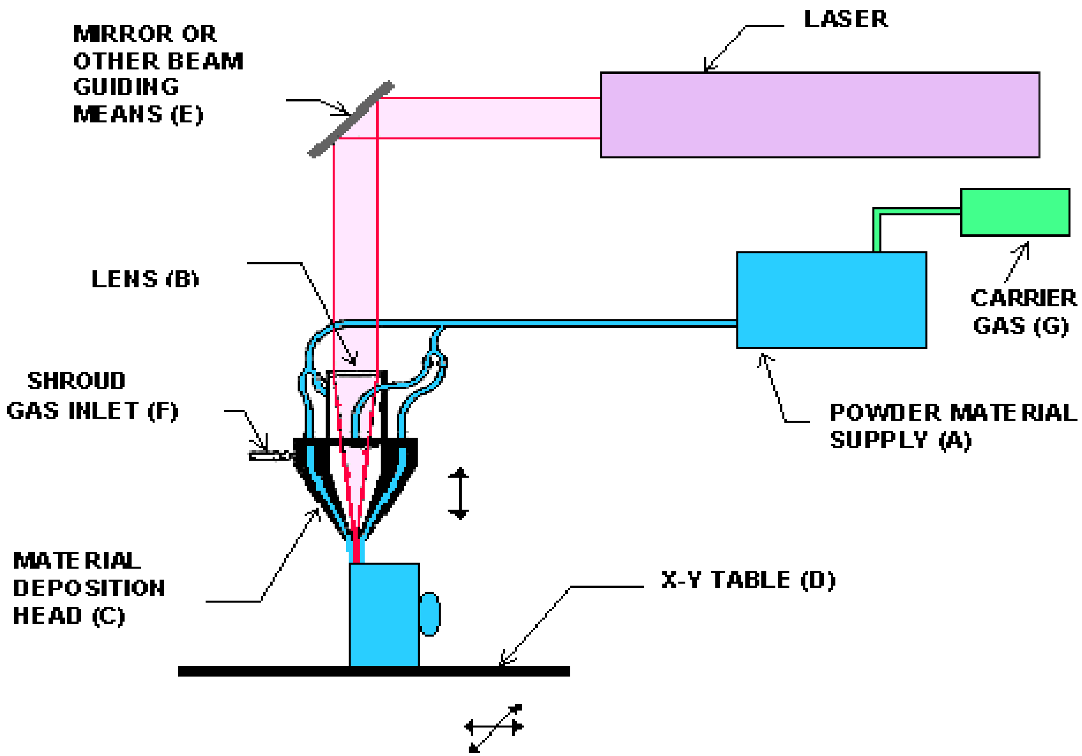

koekstruzja : rdzeń—30 % obj. nanokrzemionka / PEG200DA/ 4-HBA, osnowa—41.5 % obj. Al_2O_3/ PEG200DA/ 4-HBA
100 µm

AGH Laboratory of Laser Processing of Ceramic Materials

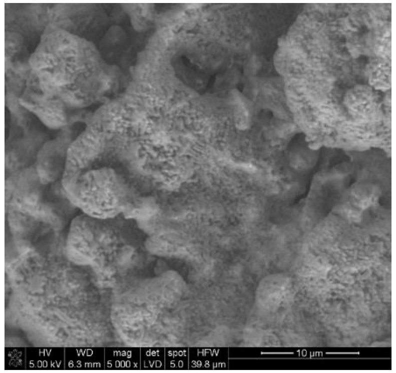
• Hybrid apparatus for Selective Laser Sintering, laser cladding, surface ablation, welding, cutting and SHS reactions

• JK2000FL equipped with ytterbium doped wire fiber

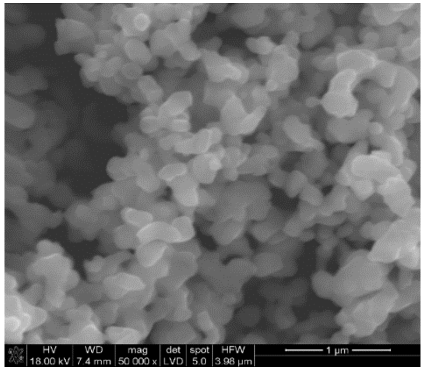
•Laser beam with wavelength of 1063[nm] ± 10[nm]



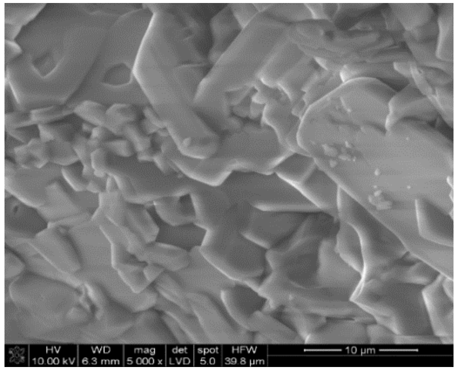
$K_{1-x}Na_xNbO_3$ (KNN) synthesis
solid state recation



K_2CO_3



Nb_2O_5



Na_2CO_3

Rotary milling for 12 hours or dry homogenization

solid state synthesis of $K_{0,5}Na_{0,5}NbO_3$ by heat treatment

DTA and XRD

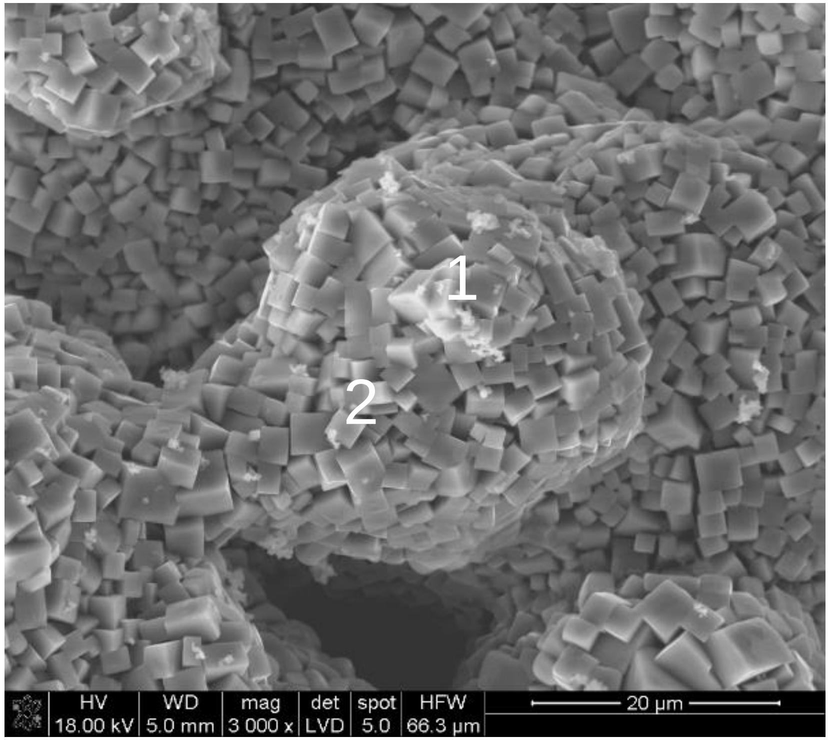
analysis of obtained powders

SEM and EDS

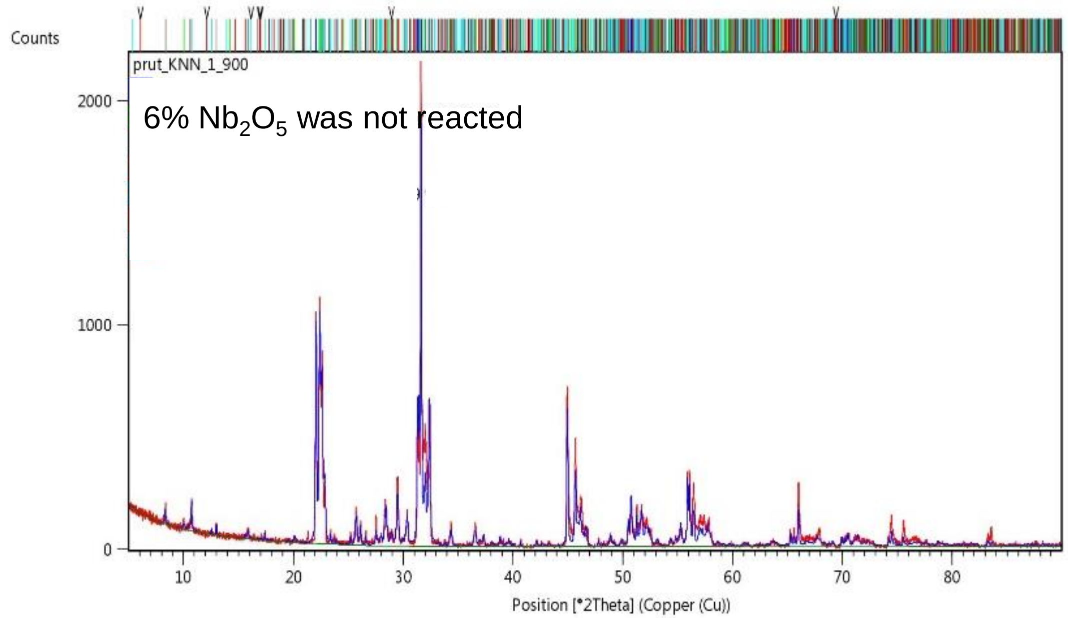
analysis of obtained powders

$$K_2CO_{3(s)} + Na_2CO_{3(s)} + 2Nb_2O_{5(s)} = 4K_{0,5}Na_{0,5}Nb_{(s)}+2CO_{2(g)}$$

$K_{1-x}Na_xNbO_3$ (KNN) synthesis solid state recation at 900ºC



Morphology by SEM

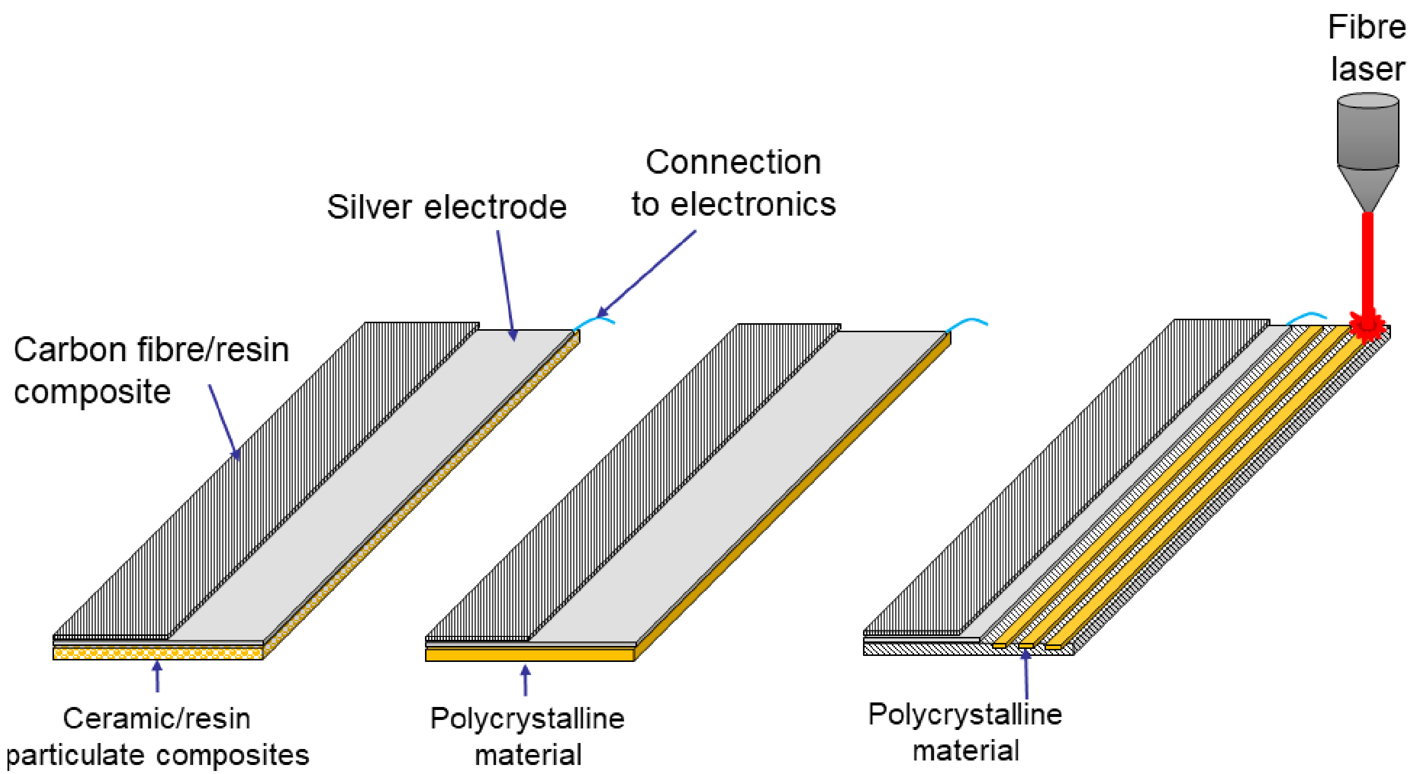


XRD analysis

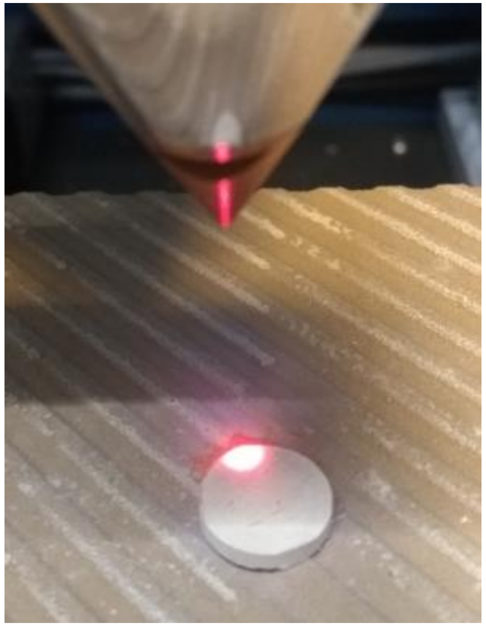
Point	Mass content [%]			
	O	Na	Nb	K
1	24,67	5,29	52,30	3,02
2	16,01	5,92	56,03	3,29

EDS analysis

Additive Manufacturing of (KNN) and PZT piezoelectric samples



Additive Manufacturing of (KNN) and PZT piezoelectric samples



PZT tested sample

PZT Selective Laser Sintered

KNN Selective Laser Sintered

Conclusion:

Additive Manufacturing is very useful perspective technology for shaping different materials

Thank you for your attention