Fraunhofer ISC
TU Clausthal
Friedrich-Schiller-Universität Jena
BAM

Sicherheit in Technik und Chemie

Bundesministerium für Bildung und Forschung

MATERIALDIGITAL

GlasDigital

GlasDigital - Data Driven Workflow for Accelerated Glass Development

Abschlusstreffen 21.06.2024

www.bam.de

GlasDigital Team
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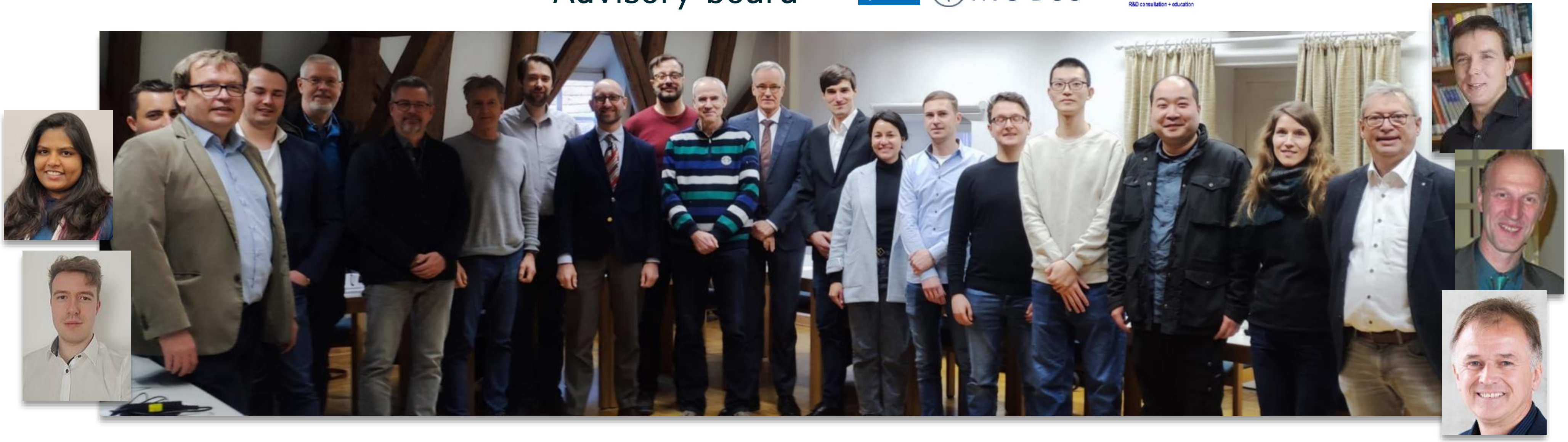
Advisory board — SCHOTT, HVG-DGG, uniglassAC GmbH

Overview

- Motivation
- Concepts, ideas
- Cooperation
- Challenges



Bold = Applicants

Glasses
Key components for advanced technologies


www.osram.de


www.scheizeldach.de/erneuerbar.html


www.DLR


www.isargfk.de


https://fuelcellmaterials.com

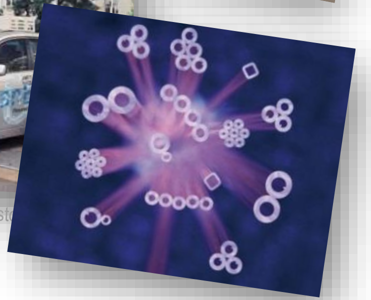

www.gerresheimer.com

PV, OLED, photo- electrochromics, electronics/ opto-electronics, LTCC, fibres, precision components, bio/dental glass, optics, pharma, nuclear waste, seals and solders, pasts, H_2-barrier (tanks)…

- Continuous and widely adjustable properties
- Unique shaping techniques

☺ **Ideal "tailored" material partner**


http://www.kfz.net/news/wasserst


http://www.hilgenberg-gmbh.de

Challenge
Glass design

☹ **Infinitely** variable composition

10^{52} compositions from 1mol% step combinations of the 80 “friendly” elements [ZanCou04]

☹ Complex **property profiles**

☹ Too little **data**, no **process** data

E.g., **SOFC glass seals**:
- Thermal expansion
- Viscosity- and crystallization
- Stable micro structure at 850°C for 80.000h,
- Adhesion ⇔ corrosion

https://i0.wp.com/www.h2-international.com/wp-content

☹ Manual **melting**

- Time consumption
- Dangers
- Poor reproducibility..

[ZanCou04] Zanotto Coutinho JNCS 347 (2004) 285

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Digital infrastructure for accelerated glass development

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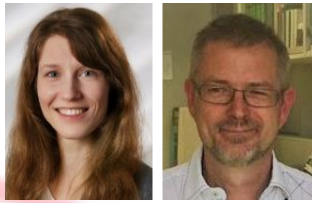
System control

BAM

Test melts



Inline sensors
HT*-Analysis

Fraunhofer ISC

ML* data mining
Glass design tools

Robotic glass melting

TU Clausthal

Image analysis

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Glass ontology
ML property modelling

FRIEDRICH-SCHILLER-UNIVERSITÄT JENA

Process modelling
(Digital twin)

Use case system
Na_2O-Al_2O_3-B_2O_3-SiO_2

*HT = High throughput *ML = Machine learning

Robotic Glass Melting System

Melting furnaces

Crucible (re-)filling

Cooling furnace (old)

Batch container

Batch dosing

Lids for mixing or crucible filling

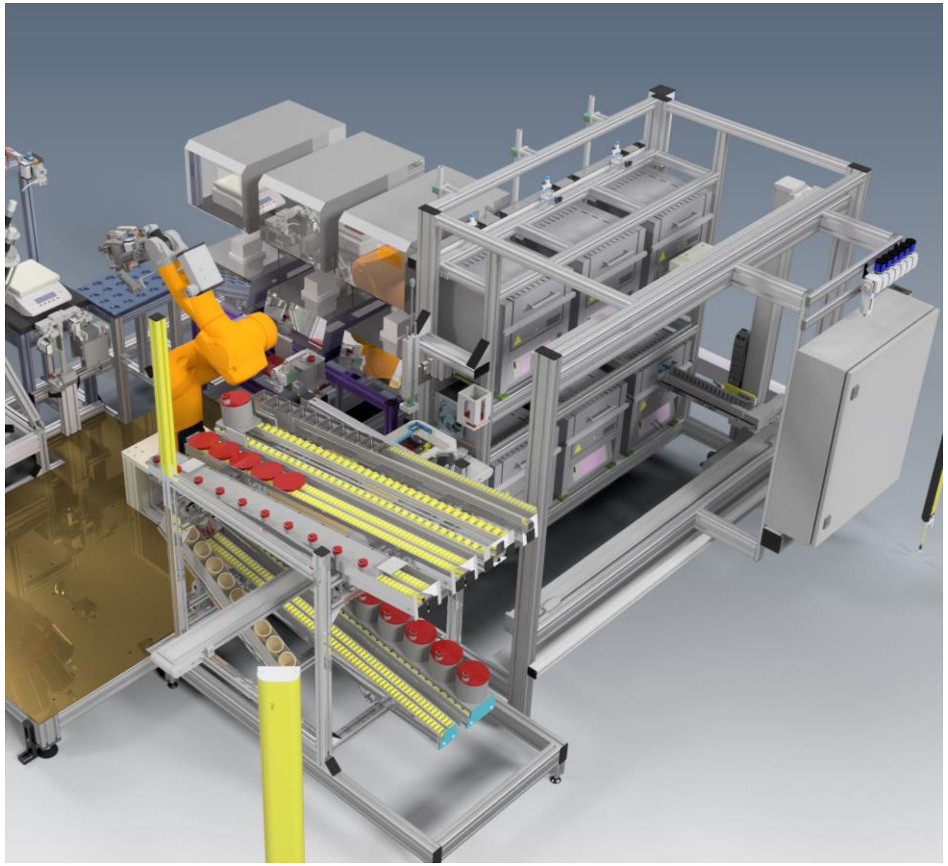
Robotic Glass Melting System

New casting and cooling system (since 7/23)

Fraunhofer ISC | BAM

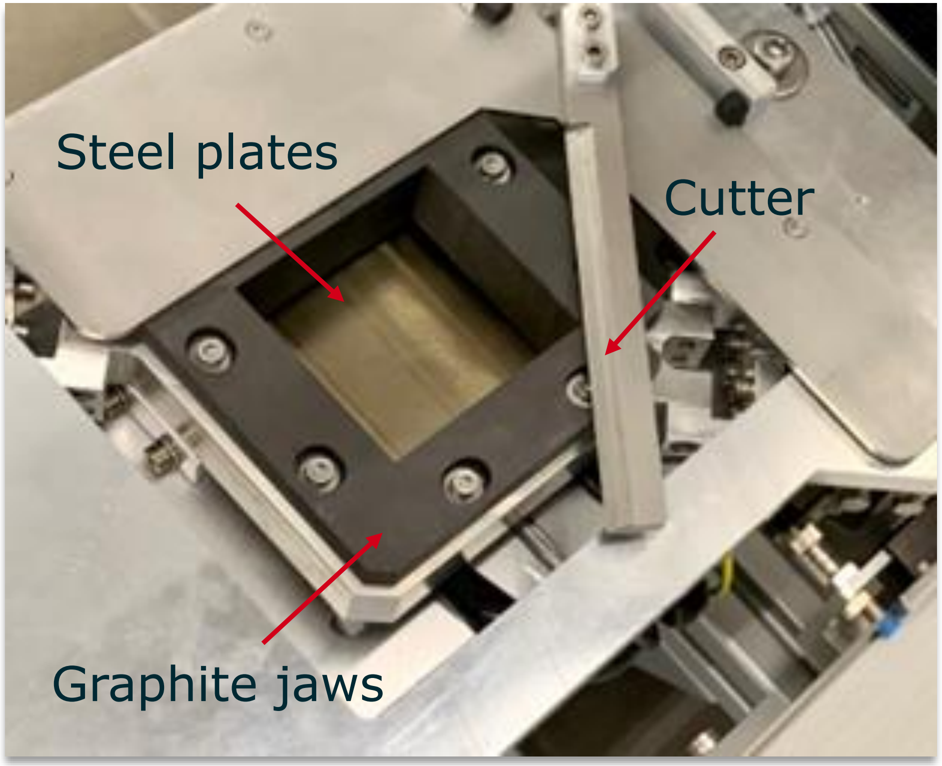
Casting on steel plates

6 Cooling furnaces



Robotic Glass Melting System

Casting and cooling system 2.0 (since 7/23)

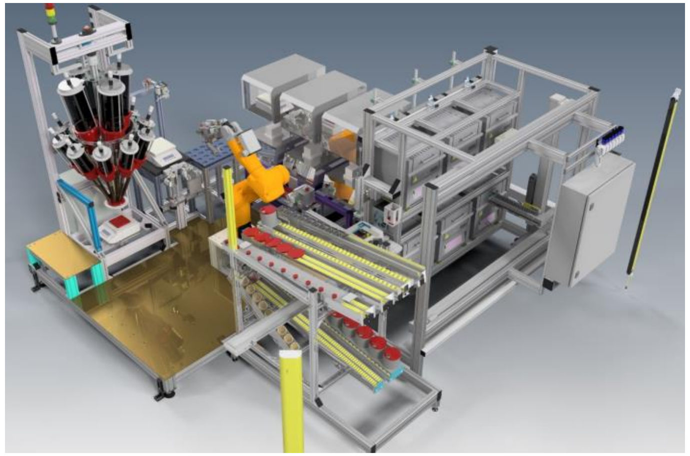


1.3 x time lap

Robotic Glass Melting System

Process data

- ☺ High-throughput
- ☺ Reproducibility
- ☺ **Process parameter** stored



"Experience"

Process protocol (json)

{"date": "2022-07-02 16:11:04.061",
"recipe": "N115A15B615S255",
"material: ": "leer", "setpoint (g)": "0.0","material: ": "test", "setpoint (g)": "0.0","material: ": "leer", "setpoint (g)": "0.0","material: ": "SiO2", "setpoint (g)": "11.47","material: ": "CaO", "setpoint (g)": "0.0","material: ": "MgO", "setpoint (g)": "0.0","material: ": "SiO2 - Millisil", "setpoint (g)": "0.0","material: ": "Na2CO3", "setpoint (g)": "9.12","material: ": "Optibor TG (H3BO3)", "setpoint (g)": "0.0","material: ": "Al2O3", "setpoint (g)": "1.14","material: ": "-", "setpoint (g)": "0.0","material: ": "B2O3", "setpoint (g)": "33.01","material: ": "K2O", "setpoint (g)": "0.0","material: ": "ZrO2", "setpoint (g)": "0.0",",
"number melt": "1", "total crucible (g)": "35.0", "setpoint refill (g)": "0.0", "oven number": "2",
"premelt heating rate (°K)": "20.0", "premelt temperature (°C)": "1100", "premelt holding time (min)": "15.0",
"main melt heating rate (°K)": "20.0", "main melt temperature (°C)": "1200", "main melt holding time (min)": "60.0",
"fritten": "FALSE",
"cool oven start time (min)": "0.0", "cool oven start time temperature control (min)": "30.0", "cool oven start temperature 1 (°C)": "700", "cool oven cool rat 1 (°K)": "3.0", "cool oven start temperature 2 (°C)": "300", "cool oven cool rat 2 (°K)": "10.0", "cool oven target temperature (°C)": "50"}
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{"timestamp": "16:13:45.489", "Processing ends job no.": "2"}
{"timestamp": "16:13:46.507", "start job no.": "3", "ID": "185", "recipe": "N115A15B615S255"}
{"timestamp": "16:13:52.119", "mat.no.": "4", "mat.": "SiO2", "setpoint (g)": "11.47", "actual value (g)": "0.0"}

Glass*Digital*
Digital infrastructure for accelerated glass development

Fraunhofer ISC
ML* data mining
Glass design tools

Fraunhofer ISC
System control

BAM
Test melts

Inline sensors
HT*-Analysis

TU Clausthal
Image analysis

Robotic glass melting

HT Raman

Friedrich-Schiller-Universität Jena
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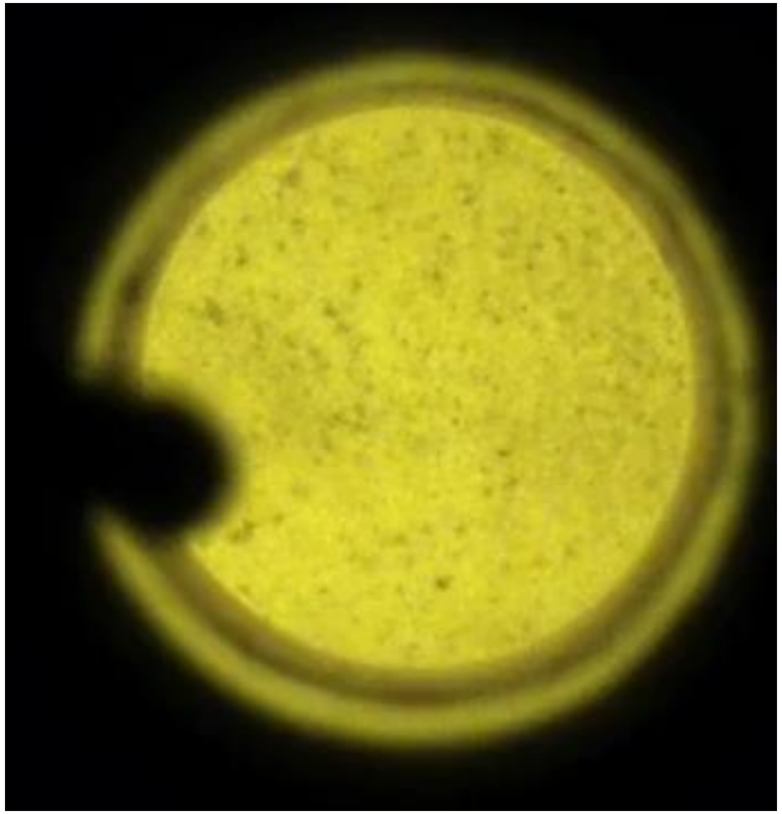
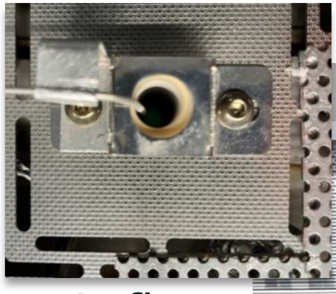
Friedrich-Schiller-Universität Jena
Process modelling
(Digital twin)

*HT = High-throughput *ML = Machine learning

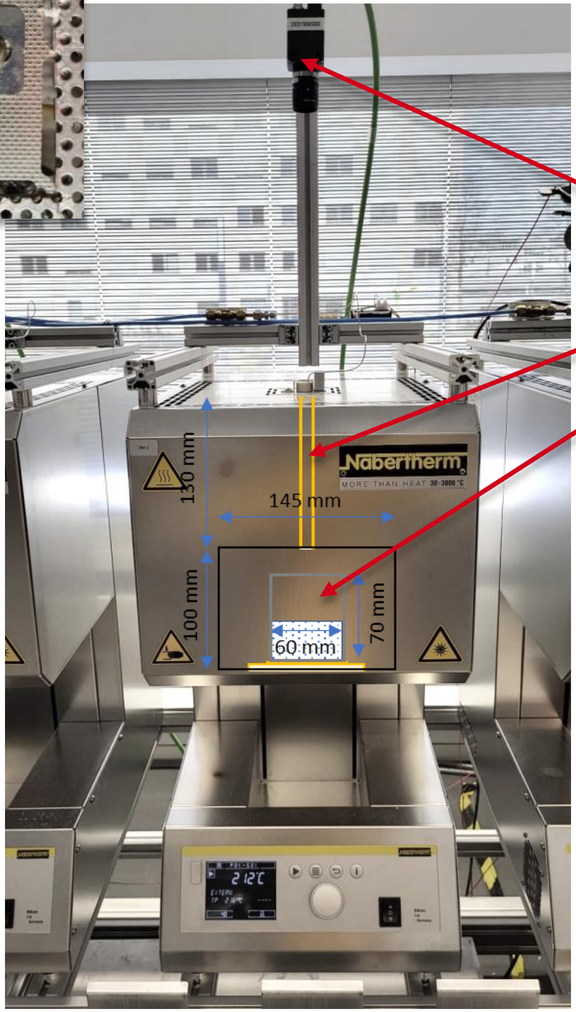
Inline Sensors
Furnace cameras



1350→1450°C 20K/min after 5th batch refill
$30Na_2O$ – $10CaO$ - $60SiO_2$
Δt = 6:23 min (10x time lap)



Air flow



Baumer VCXG-15C.I, 35 mm
Camera

Al_2O_3 tube

Crucible

- ☺ Look through the 13 x 1cm tube!
- ☹ Condensation, smoke → air flow, tube replacement

Inline Sensors

Real-time melting stage evaluation

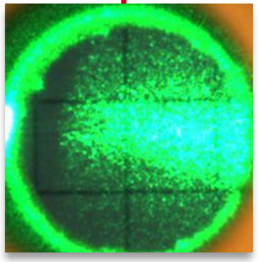
1050°C, 10 fps, Sodium alumino silicate batch, Time to melt 300 s (10 x time lap)

ML Image analysis (ResNet 34) detects different melt stages

Granules   Foaming   Fining

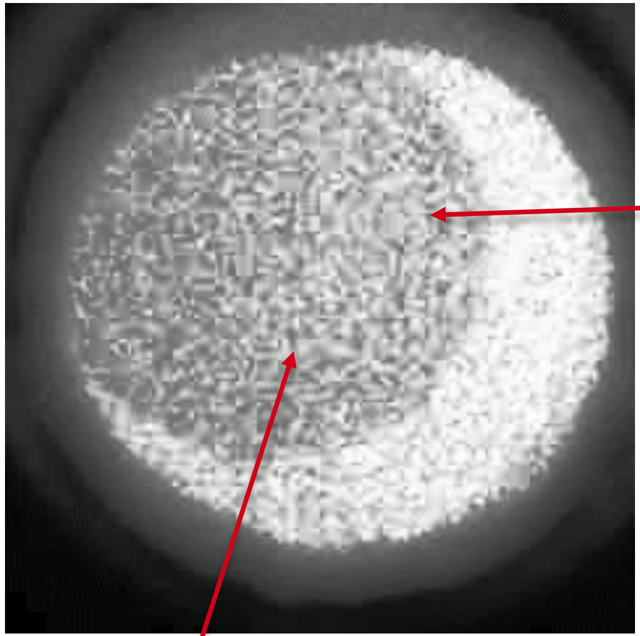
Laser light detection of foaming up

(in progress)

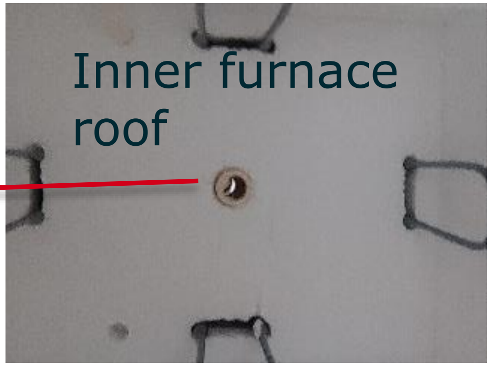


- ☺ more reliability
- ☺ more output
- ☺ less energy

Inline Sensors
Real-time castability evaluation



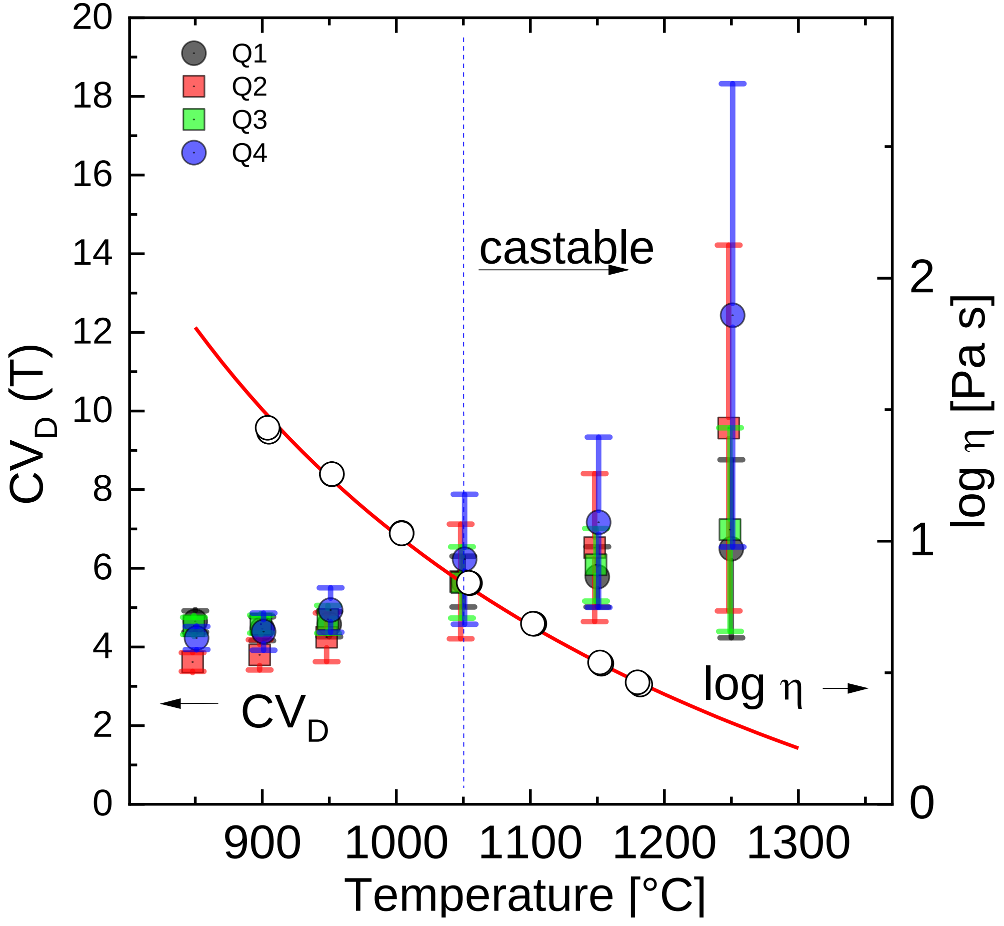
1250°C, 30 fps, Sodium alumino silicate batch, (10x time lap)



- Mechanical impacts during continuous camera observation
- Image change rate scales with viscosity

Reflection of the furnace tube

☺ Failure probability ⇩
☺ Reproducibility



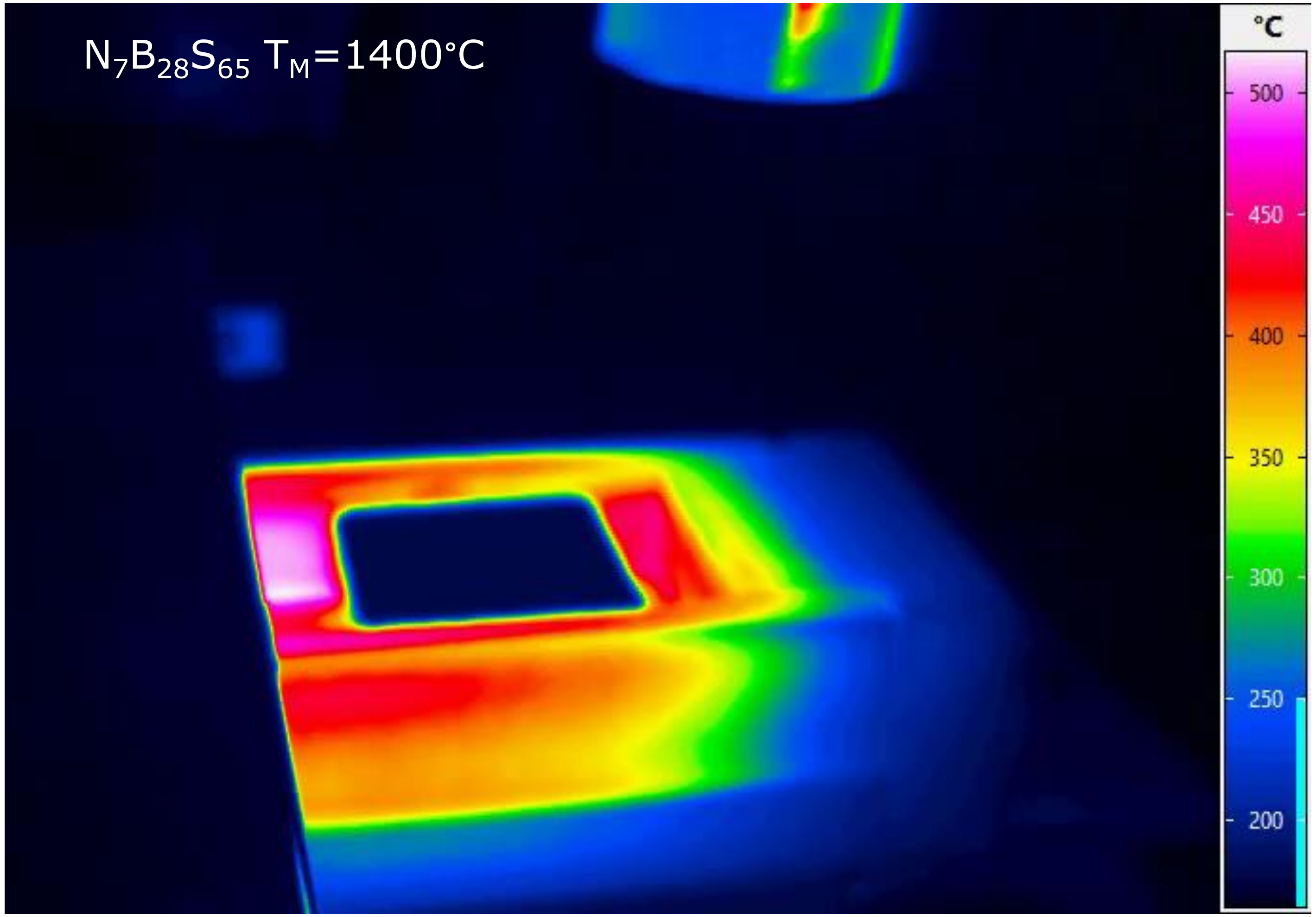
Gogula et al. Optical real-time castability check for high-throughput glass melting, Glass Europe 2 (2024)

Inline Sensors
Casting

IR camera

InfraTec

VarioCAM HDx



Casting temperature and “choreography”
→ better homogeneity

***Glass**Digital*

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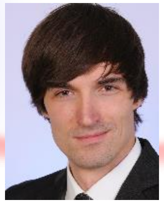
HT* Raman



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ML property modelling

Friedrich-Schiller-Universität Jena

Process modelling
(Digital twin)

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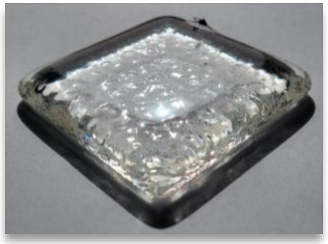
High-Throughput Analysis

Chemical composition ✓



PANanalytical Zetium Ultimate

XRF

☺ Automated system at BAM 1.4

(10min for 5 elements = 60 samples a day)

☹ Boron analysis

→ Combine with **wet chemical B** analysis

→ Combine it with **LIBS** (start in 2025)

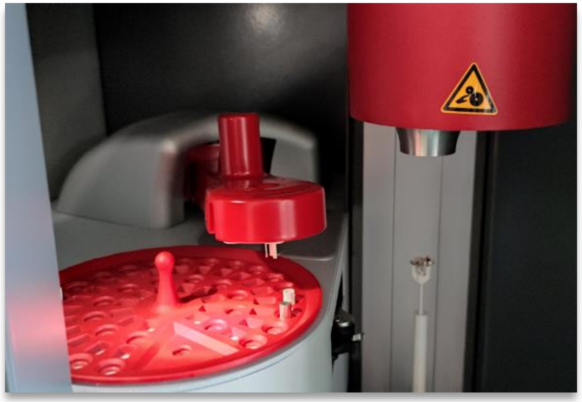
	Composition [wt%]		
	Na_2O B | A	B_2O_3 B | A	SiO_2 B | A
$N_{16}B_{10}S_{74}$	16,17 | 15,56	11,35 | 10,50	72,48 | 73,8
$N_{15}B_{35}S_{50}$	14,59 | 14,34	38,25 | 39,20	47,16 | 46,3
$N_{25}B_{25}S_{50}$	24,62 | 24,74	27,65 | 25,96	47,73 | 49,2
$N_{10}B_{40}S_{50}$	9,67 | 11,14	43,45 | 43,27	46,88 | 45,5
$N_{20}B_{30}S_{50}$	19,58 | 16,48	32,98 | 31,42	47,44 | 52,0
$N_{16}B_{10}S_{74}$	16,17 | 15,59	11,35 | 10,94	72,48 | 73,1
$N_{16}B_{10}S_{74}$	16,17 | 14,88	11,35 | 10,71	72,48 | 74,2

≈ 1.5 mol% accuracy (including $\Delta m=0.05g$ dosing station)

B Batch
A Analysis

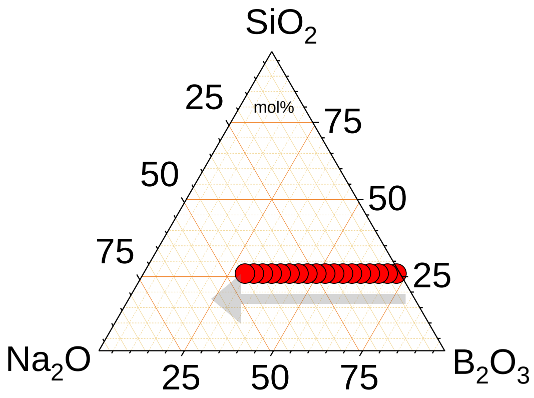
High-Throughput Analysis

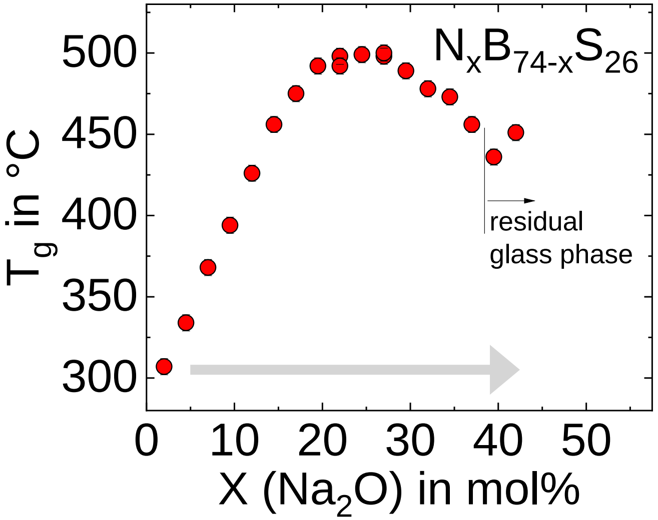
Glass transition temperature T_g (✓)

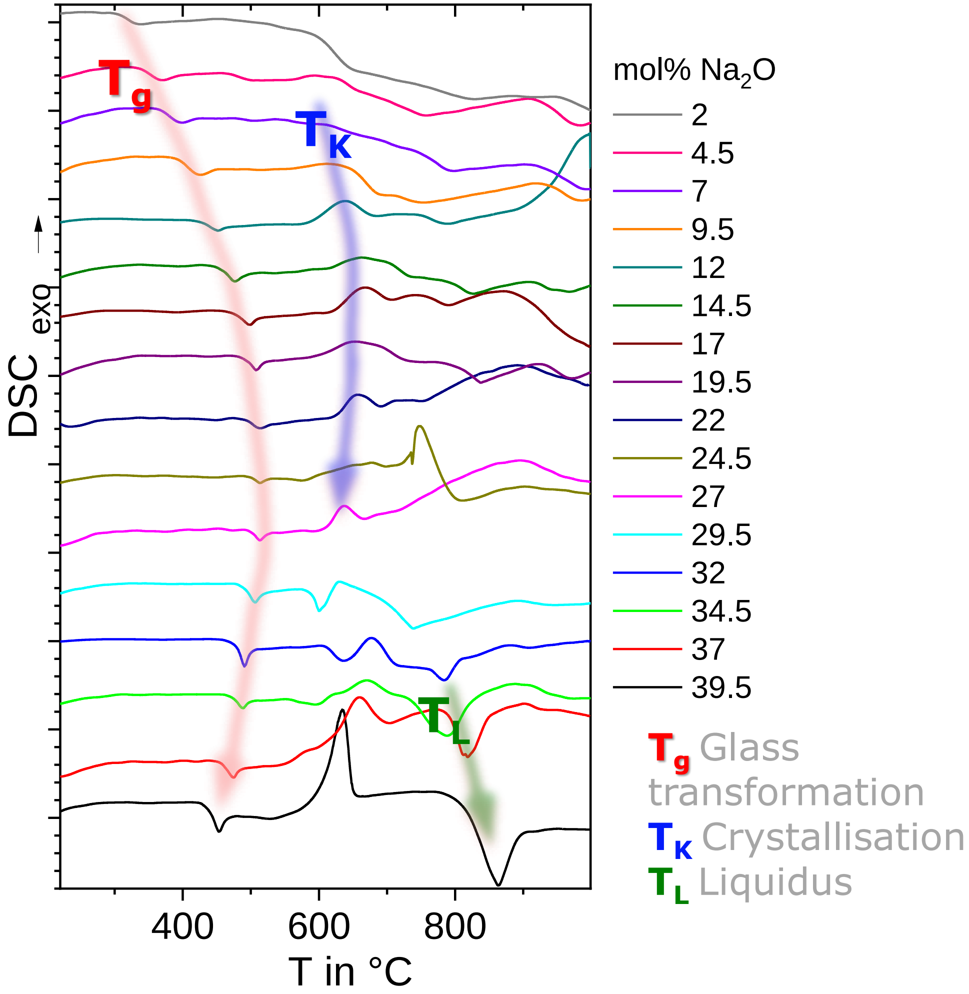


DSC Setaram Themys One+

- ☺ Autosampler for 30 samples (series: 15x6h = 4 days)
- ☹ No automated data processing

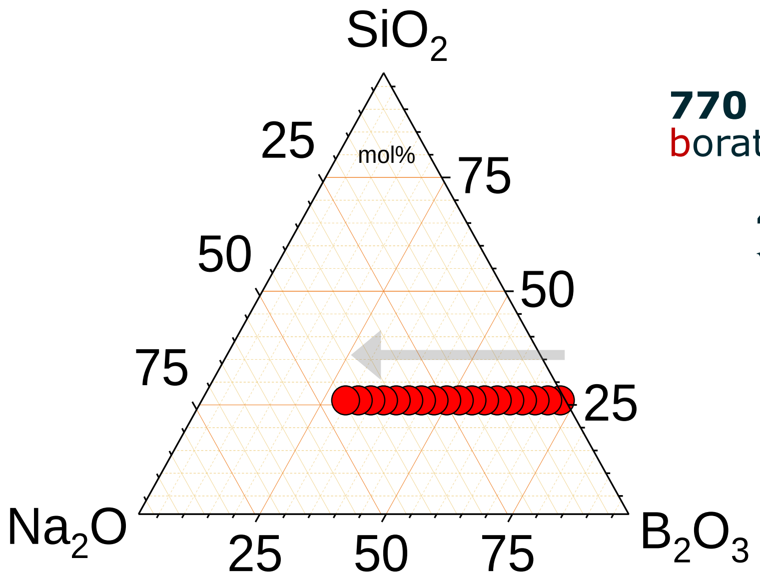






High-Throughput Analysis
Structural investigation (✓)

Confocal Raman microscope Renishaw inVia™



Test example
$N_xB_{74-x}S_{26}$

- ☺ 1 min per sample (😐 30 min high resolution)
- ☹ Data evaluation time-consuming

805 *B*-3membered boroxol rings $[BO_3]$

800 δSi-O-Si

930 νB-O $[BO_4]^-$

770 *B*-3membered borate rings with $[BO_4]^-$

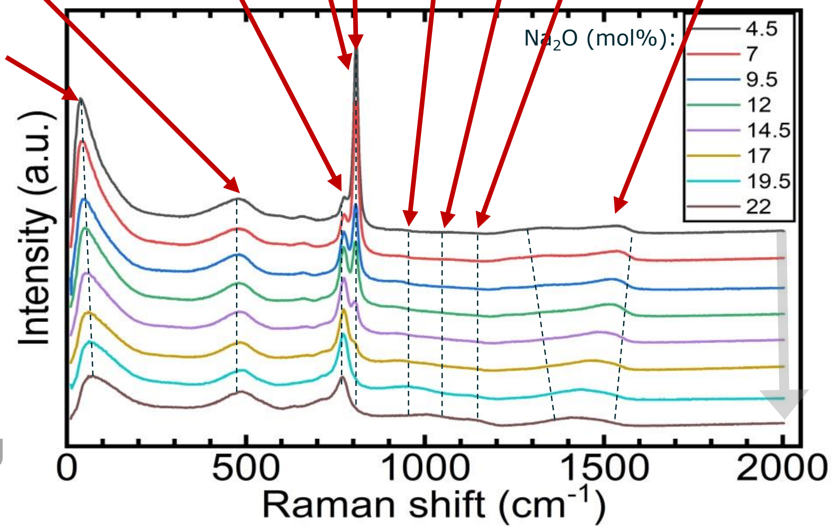
1060 ν_{as}Si-O-Si (Q4)

1140 νB-O-Si

470 mixed ν,δSi-O-Si (**MB**)

1300-1600 δB-O $[BO_3]$

BP



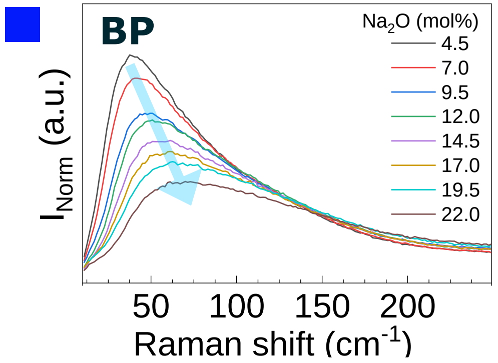
δ-Bending
ν-Stretching
B-Breathing

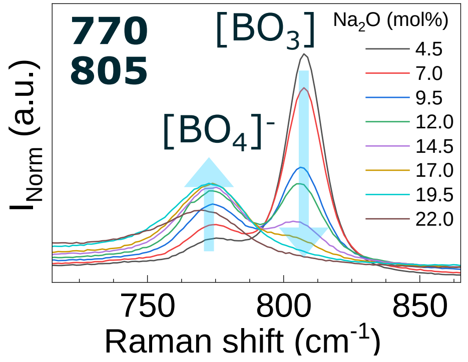
(514 nm Ar, notch filter, 8-2000 cm^{-1}, D 1.3 cm^{-1})

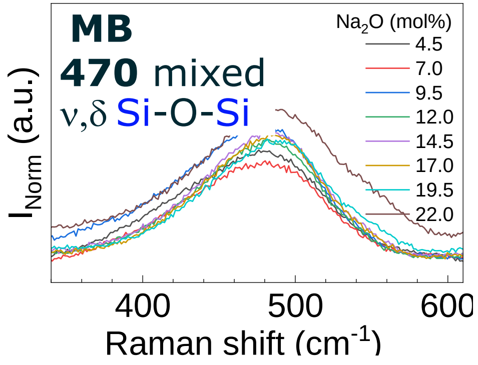
Band assignments: Winterstein-Beckmann et al., JNCS 401 (2014) 110-114 & JNCS 405 (2014) 196-206

High-Throughput Analysis

Automated Raman data processing ✓

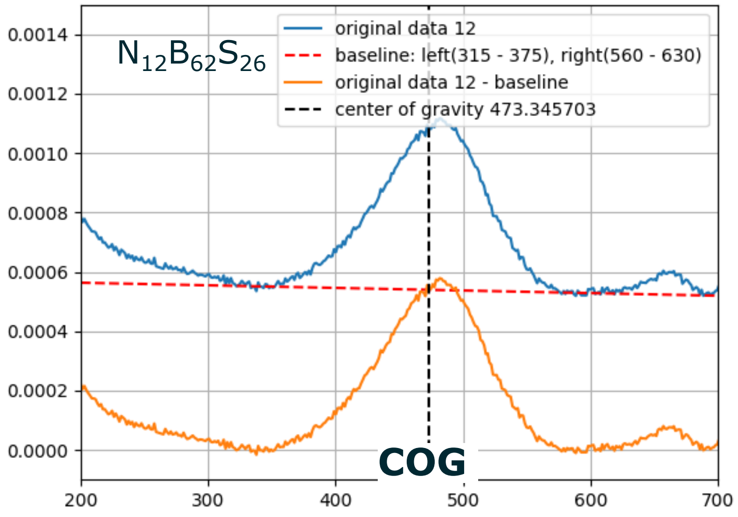


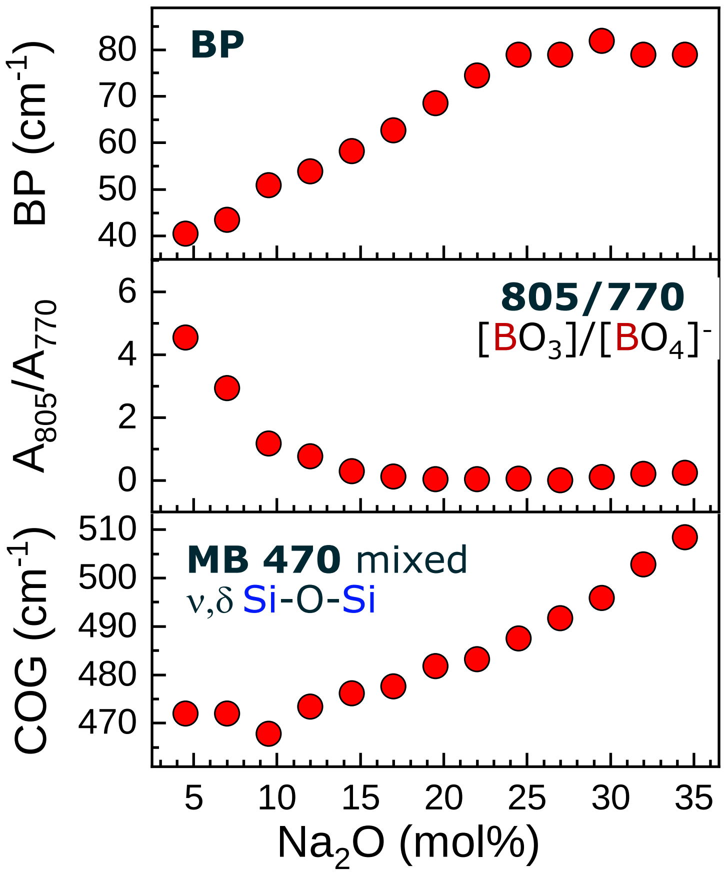




Python script:

- Data input (.txt files)
- Spectra normalization
- Band assignments
- Base line correction
- Peak fits, COG examination
- Feature extraction





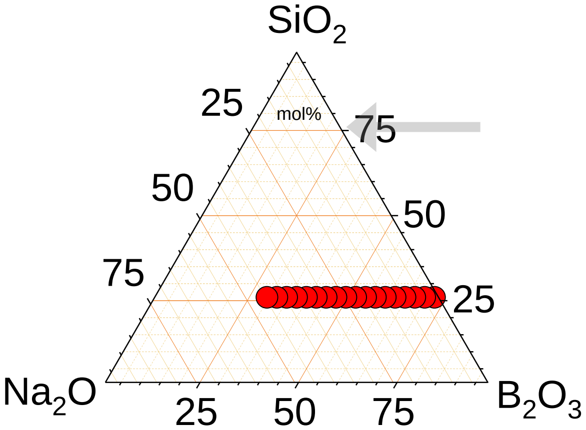
☺ Data evaluation within seconds !

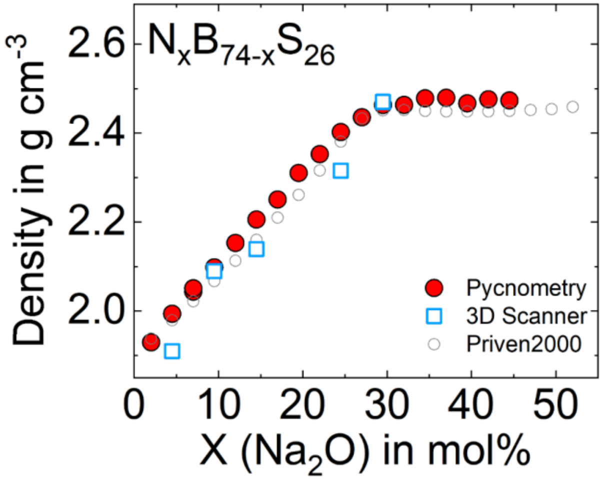
High-Throughput Analysis
Density (?)

Pycnometry
- ☺ Accuracy (0.02%)
- ☺ 10 min (T = const.)
- ☹ Fully manual

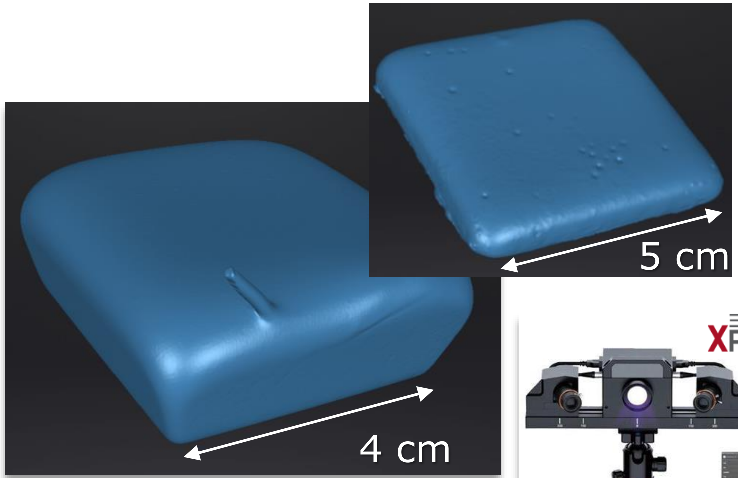
(No sampler, too difficult for cobots)

Anton Paar
Ultrapyc 5000





3D Scanner
- ☺ Fast measurement (Scan 1min)
- ☺ Easy automatable
- 😐 Less accurate for large samples
  (Δz = 35µm → $\Delta V_{5x5x2cm}$ = 0.3%) (→0.1%)
- ☹ Not yet automated data processing



Shining TransscanC*

https://www.newegg.com

→ Money - capacity - accuracy

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Process modelling
(Digital twin)

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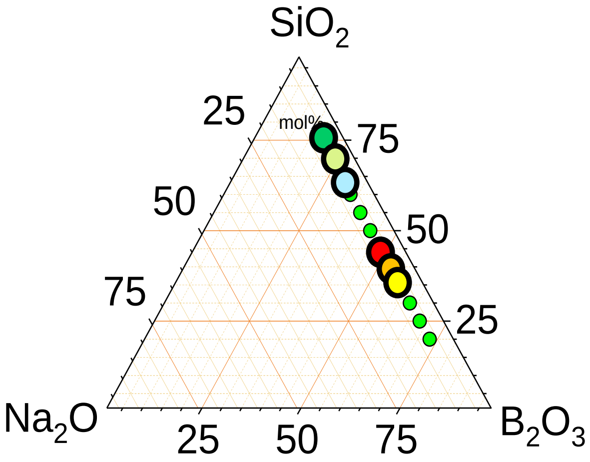
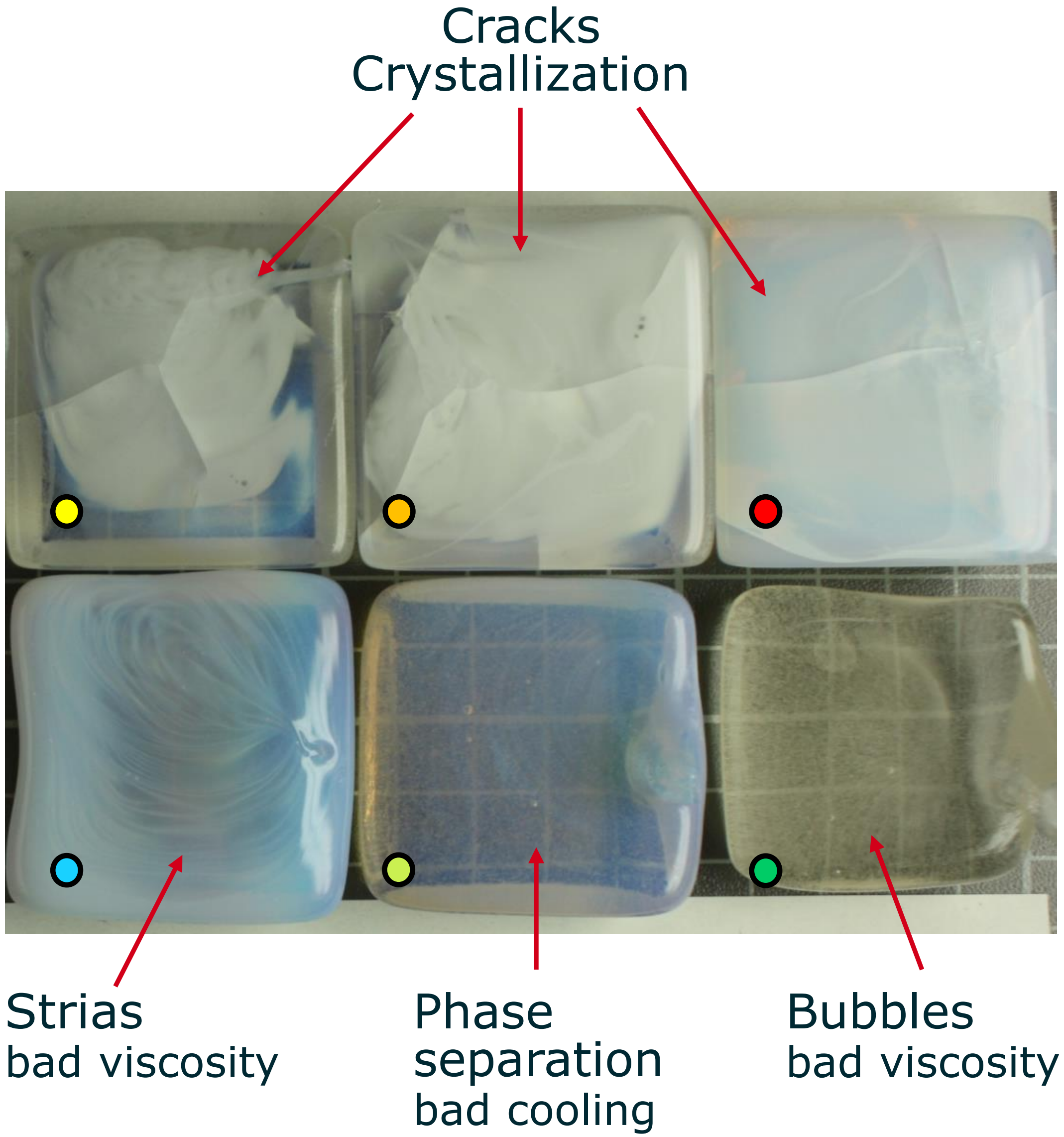
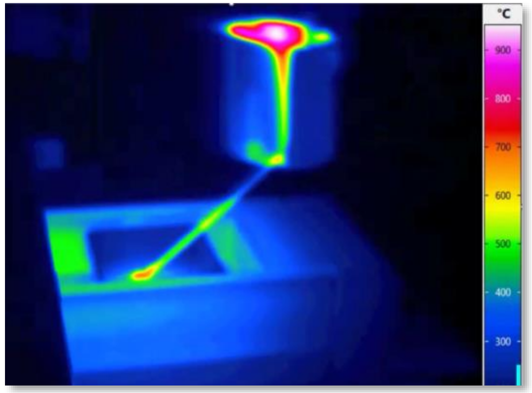
Process Modelling

Digital twin







Prediction of casting & cooling parameters, casting choreography

Process Modelling
Casting

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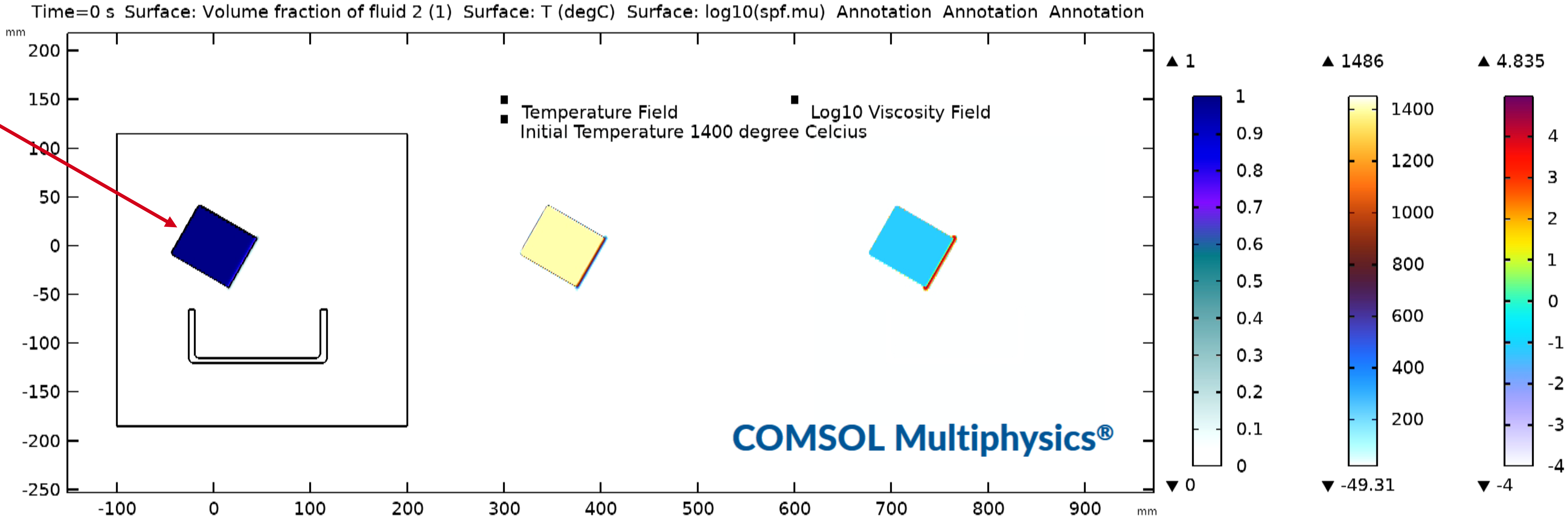
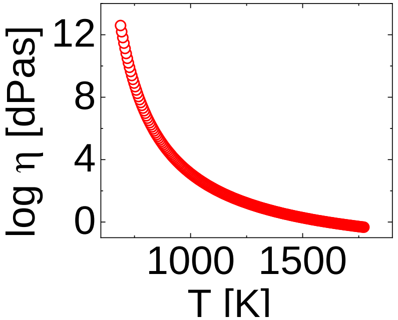
Volume Temperature Viscosity Fraction K lη/Pas

Crucible
∅ 5 cm



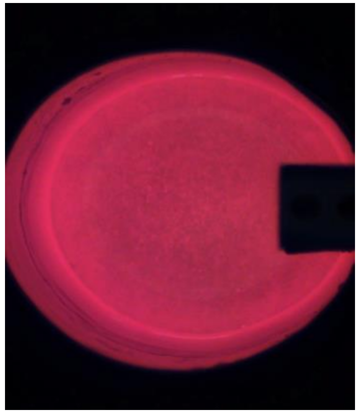
Slow motion 13x
Start at 1400°C

$N_{12}B_{62}S_{25}$ η(T) measured at OSIM, T_M=1450°C



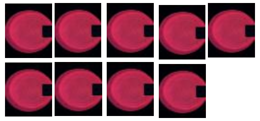
Viscosity predicted or inline measured?

Process Modelling
Inline viscosity measurement

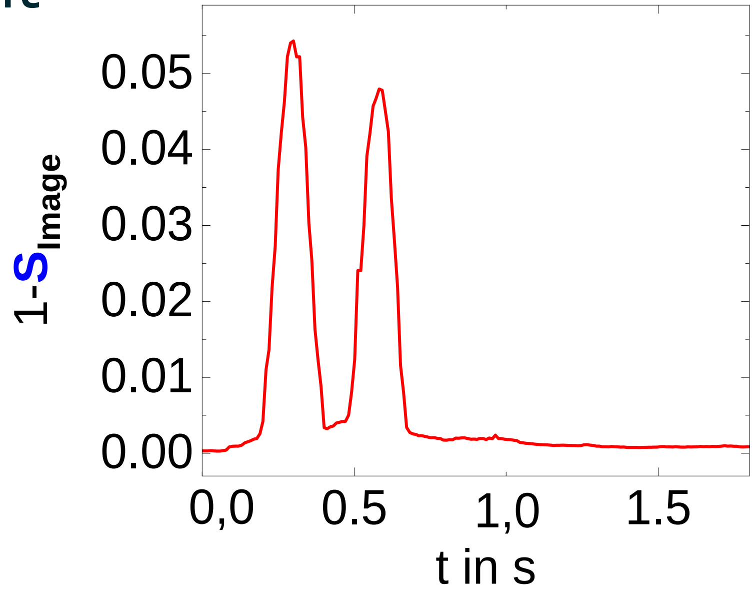


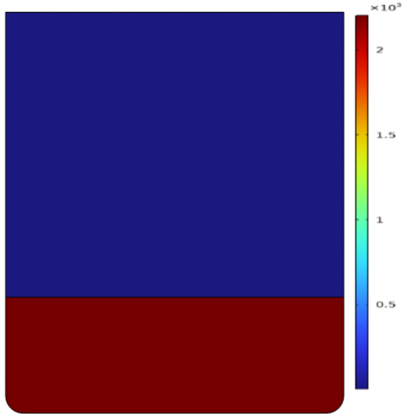
Sodium borosilicate batch
1050°C, 95 fps, Vis Camera

Crucible shaking
Image series



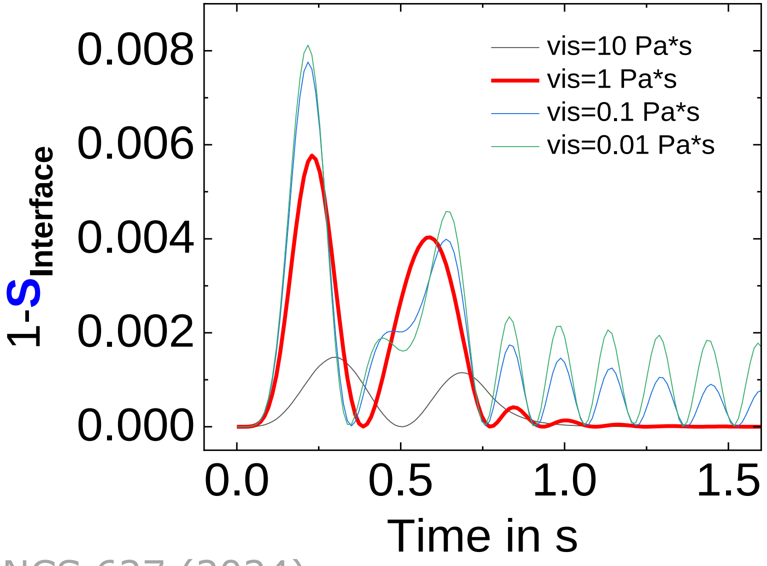
Similarity to 1st frame **S**=0…1



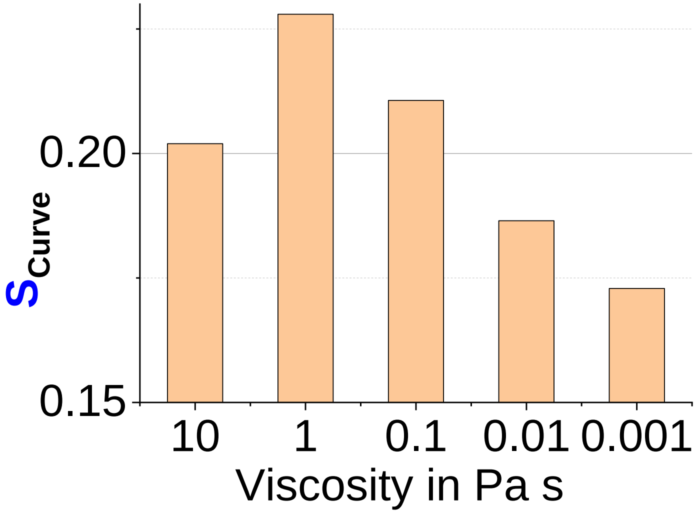


COMSOL Multiphysics®

2d-FEM:
Interface profile similarity to flat interface (CC function [1])



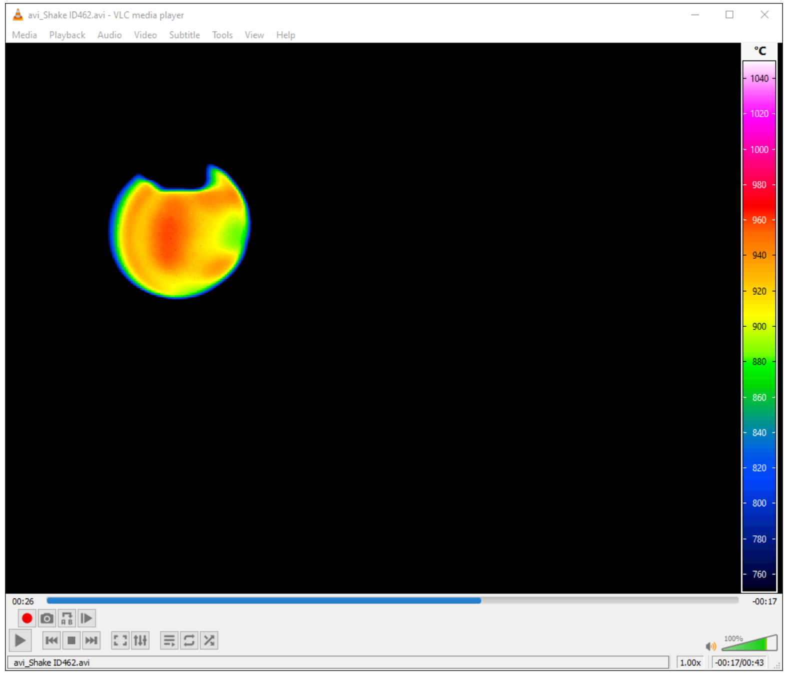
Curve similarity
(CC function [Pan24])



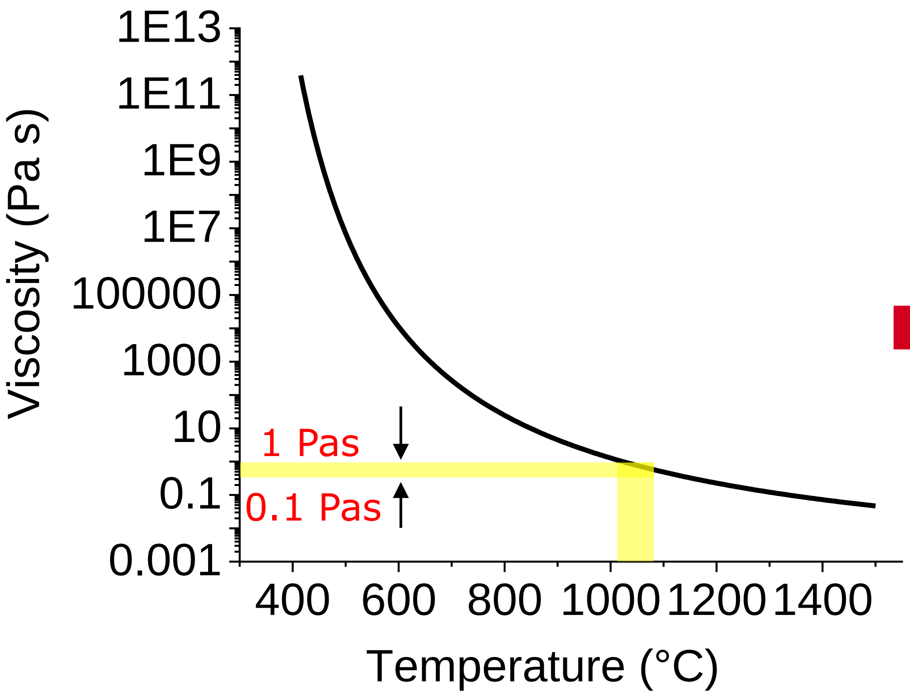
[Pan24] Pan et al., JNCS 627 (2024)

Process Modelling

Inline viscosity measurement



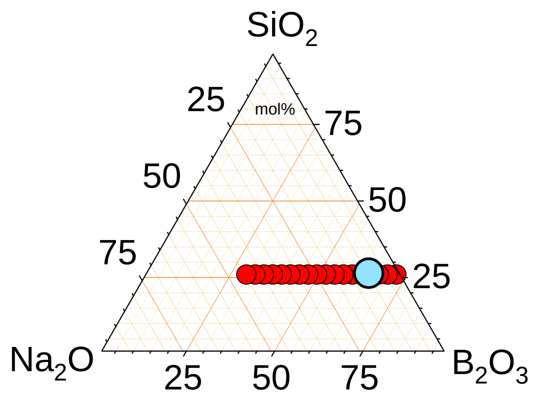
Temperature measured with IR camera

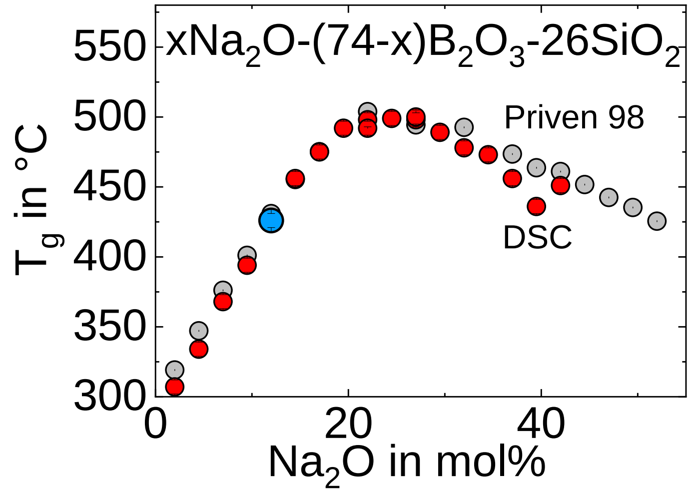


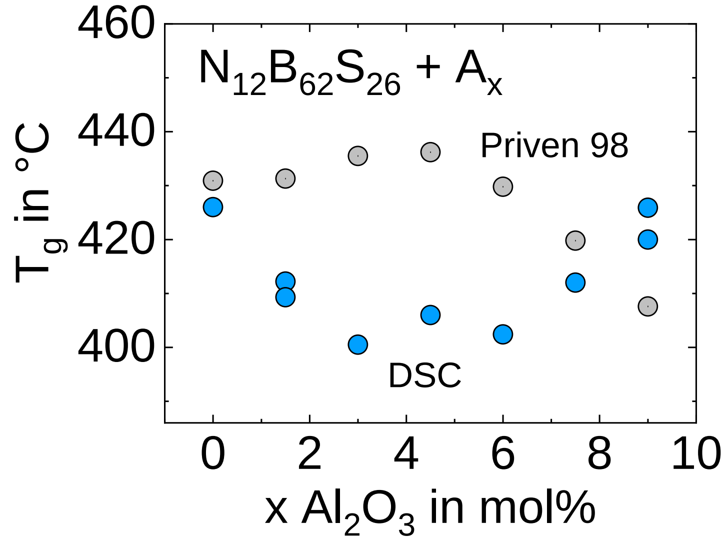
Experimental viscosity data agree with their modelled counterparts for this temperature

☺ Robust and fast inline viscosity measurement

Process Modelling
Cooling







Data

$T_{g,}$ and dT/dt_c, thermal properties,… have to be

- estimated from literature, process data, databases
- or modelled

Ontology based data space and efficient ML modelling

Glass*Digital*

Digital infrastructure for accelerated glass development

BAM

Fraunhofer ISC

System control

BAM

Test melts

Inline sensors
HT*- Analysis

Fraunhofer ISC

ML* data mining
Glass design tools

Robotic glass melting

TU Clausthal

Image analysis

FRIEDRICH-SCHILLER-UNIVERSITÄT JENA

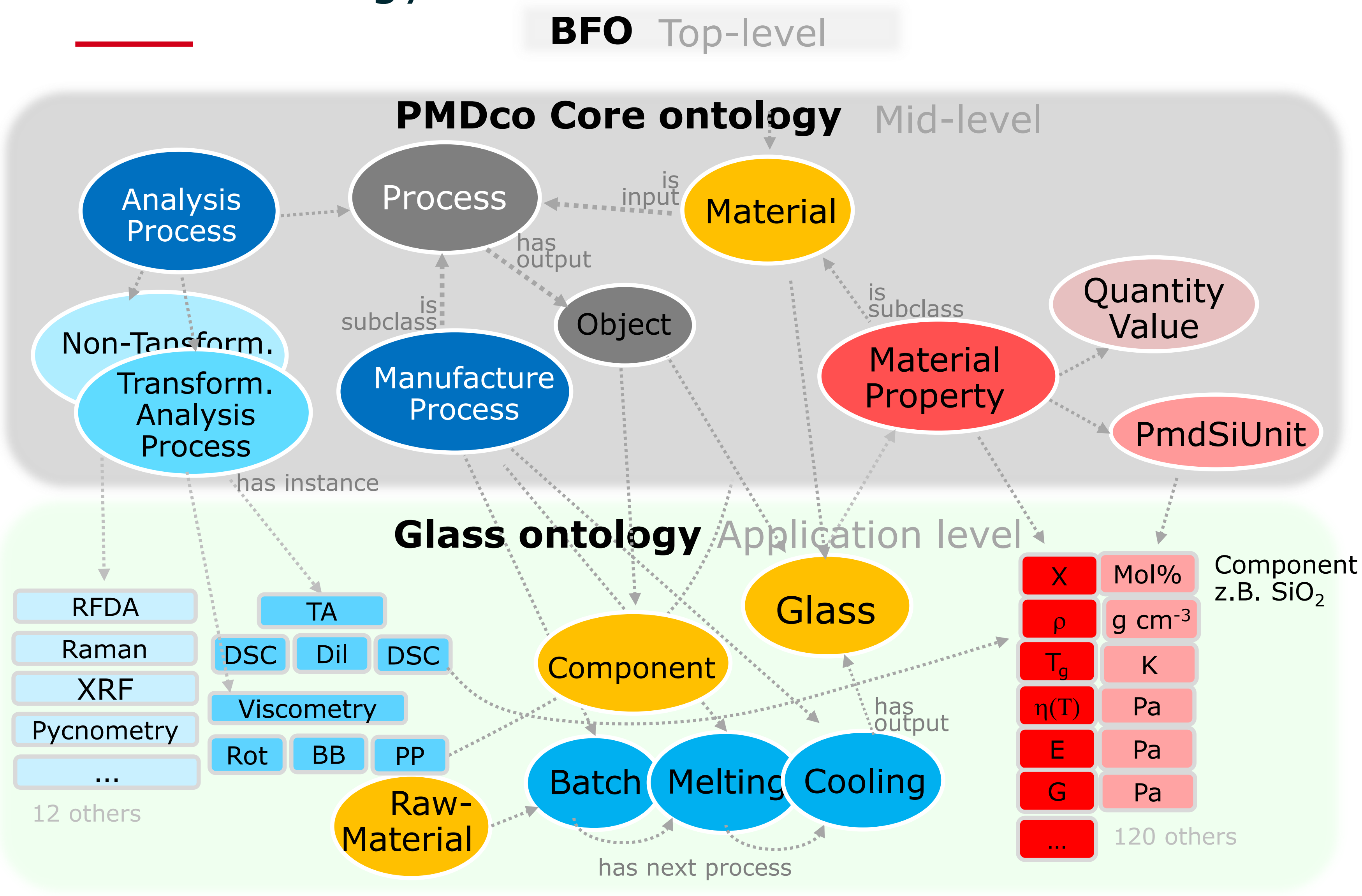
FRIEDRICH-SCHILLER-UNIVERSITÄT JENA

Glass ontology
ML property modelling

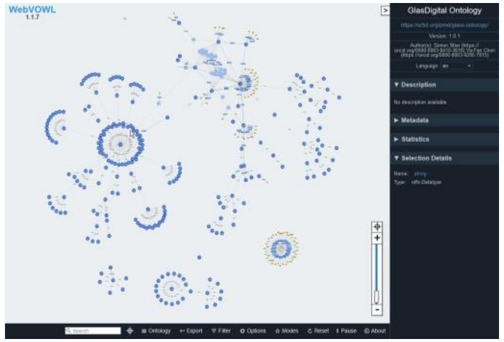
Process modelling
(Digital twin)

*HT = High throughput *ML = Machine learning

Data Space
Glass ontology



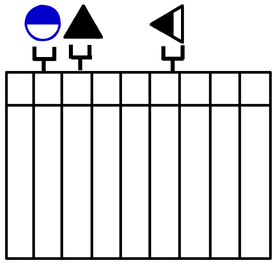
Glass ontology

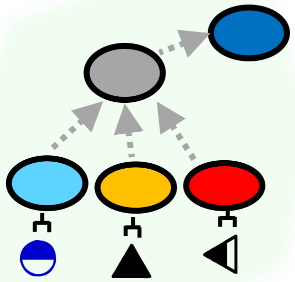


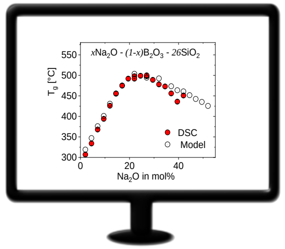
https://github.com/materialdigital/glasdigital-ontology

- ☺ Compatibility to other materials and ontologies
- ☹ Permanent adjustments

Data Space
Process pipeline







Glass data | Glass ontology | Glass- and Process design

.docx .xlsx
.json
native

Wrapper

.json
canonical

Mapper

Resource
Description
Framework

Build with
protégé

Visualising
WebVOWL

.owl* .ttl

Web
Ontology
Language

PMD App
OntoDocker

Apache
Jena
Fuseki

SPARQL*
server

*Protocol and RDF
Query language

GUI Queries
Graphical User
Interface

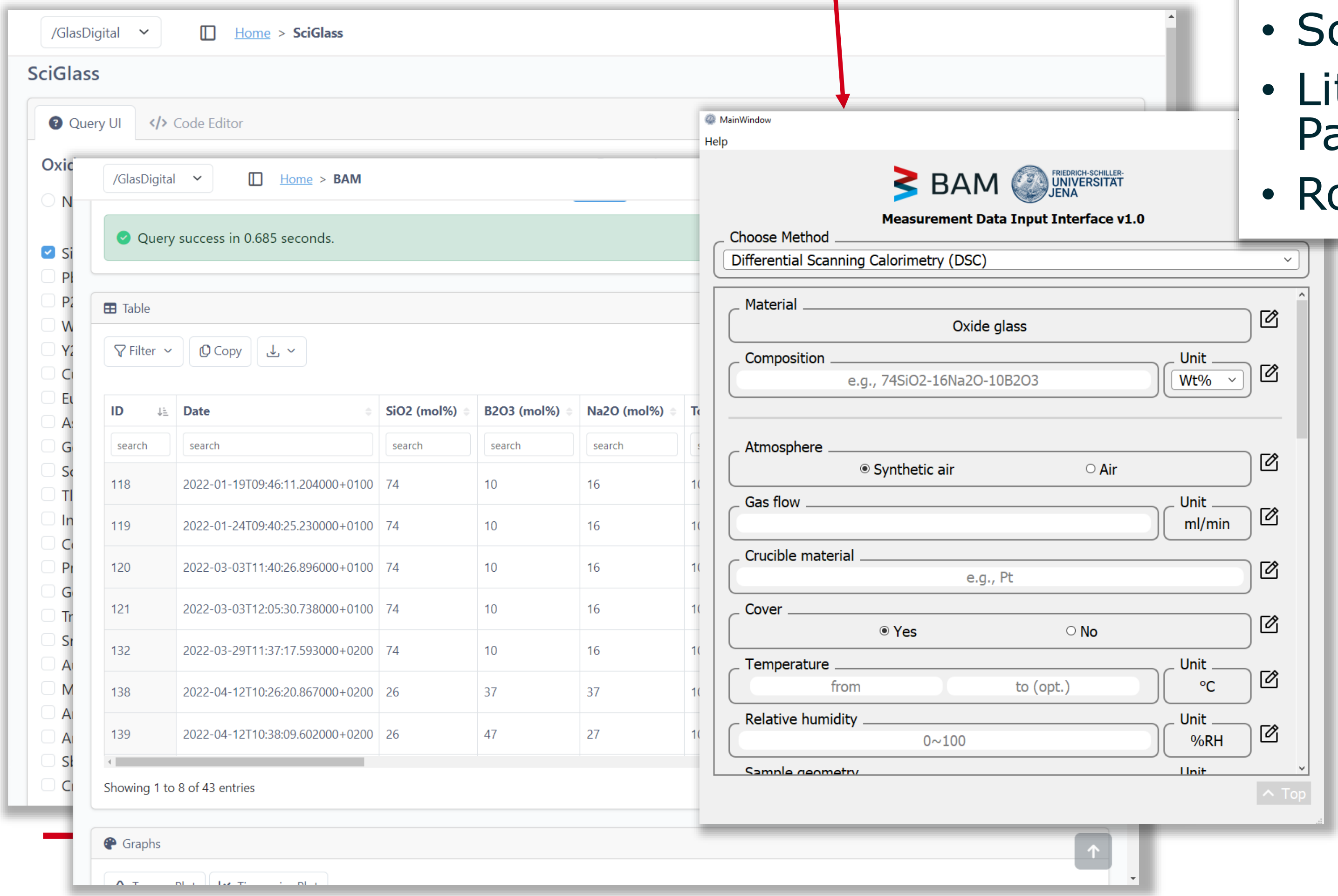
API Data upload &
delete & user
authorization
Application
Programming
Interface

https://git.material-digital.de/ya-fanchen/fast-ontodocker

Data Space
Semantic knowledge database

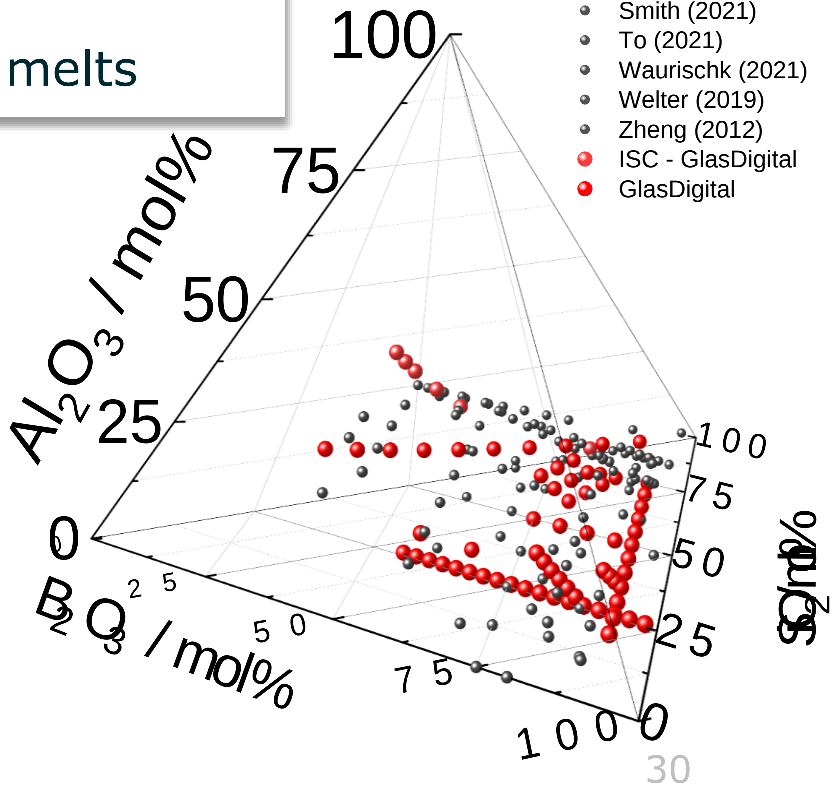
Web-based GUI
Properties and correlations

Input interface v1.0
Measurement data



Data space
~ 400.000 glasses (800 NABS)

- SciGlass
- Literature Patents
- Robotic melts

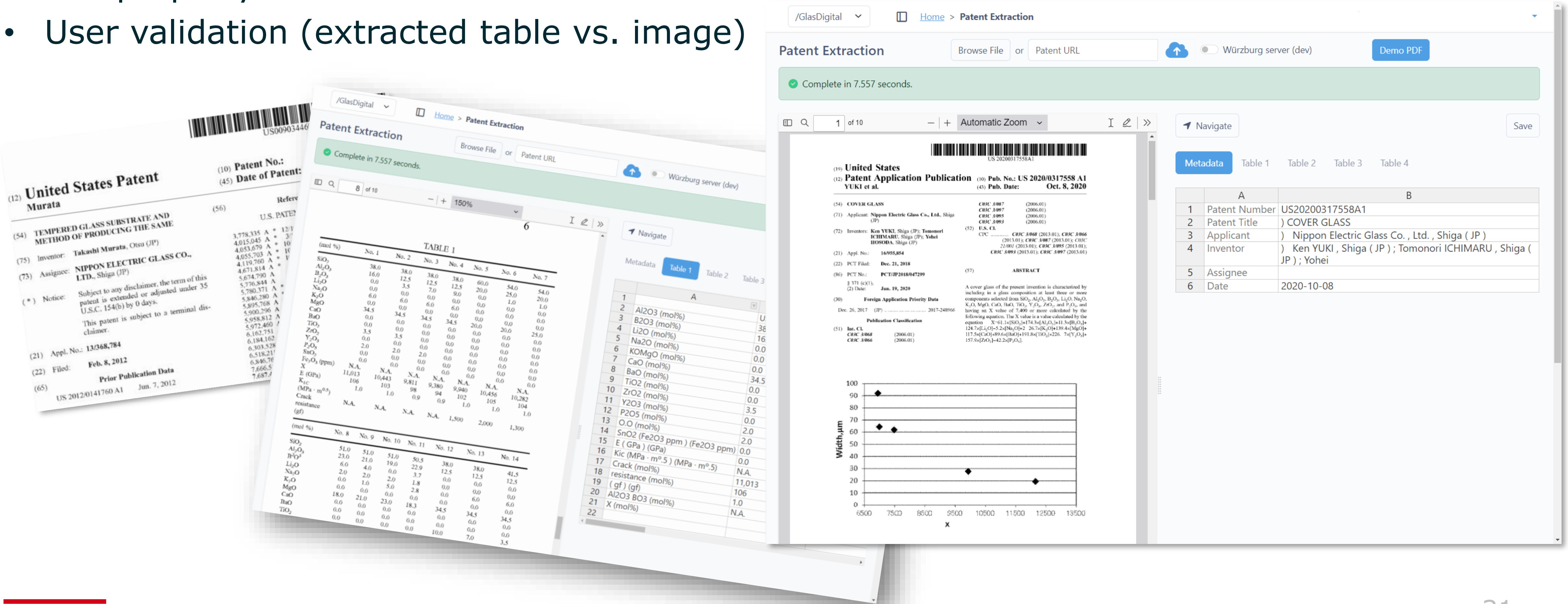


- Balzer (2019)
- Bruns (2020)
- Fuhrmann (2014)
- Januchta (2019)
- Januchta (2020)
- Krishnamurthy (2021)
- Kumar (2019)
- Limbach (2015)
- Park (2021)
- Pyrex
- Smedskjaer (2014)
- Smith (2021)
- To (2021)
- Waurischk (2021)
- Welter (2019)
- Zheng (2012)
- ISC - GlasDigital
- GlasDigital

Data Space
Data mining

Patent data extraction

- Ontology-based meta data, composition and property data from PDF text and tables
- User validation (extracted table vs. image)



ML-Modelling Glass Properties

Most efficient input parameters {descriptors}

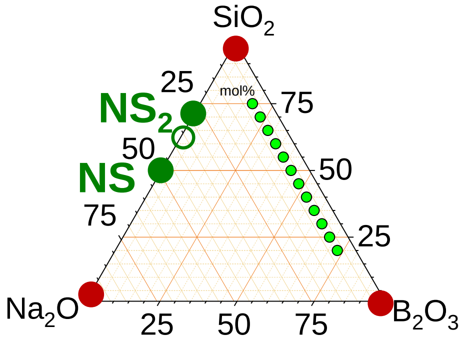
{a_i} = Atom%

{X_i} = Mol% Glass **component** (z.B. SiO_2). $P = \Sigma X_i p_i$

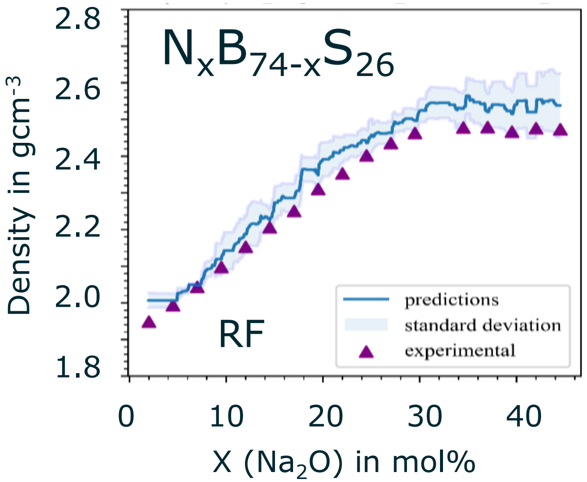
{C_i} = Mol% **constituent mineral phases** p_i of these phases in their glassy state $P = \Sigma C_i p_i$

{$P(C_i)^+$, r_i^*, u_i^*, X_i} = **DFT** calc crystal **properties** of corner compounds (●) P = ML-modelling (GPR) scikit learn PyTorch

F. Arendt et al., JACS 107 (2024)

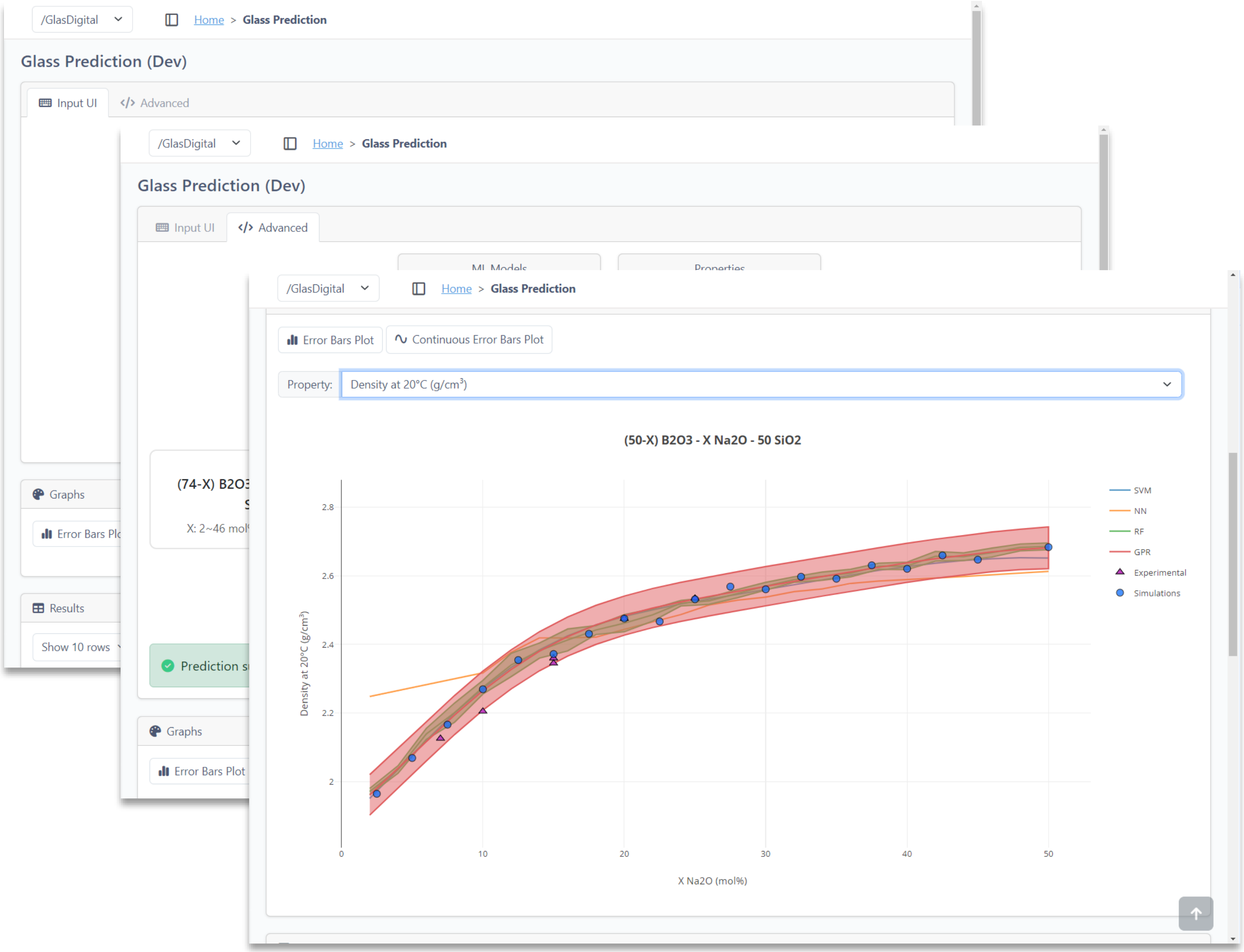


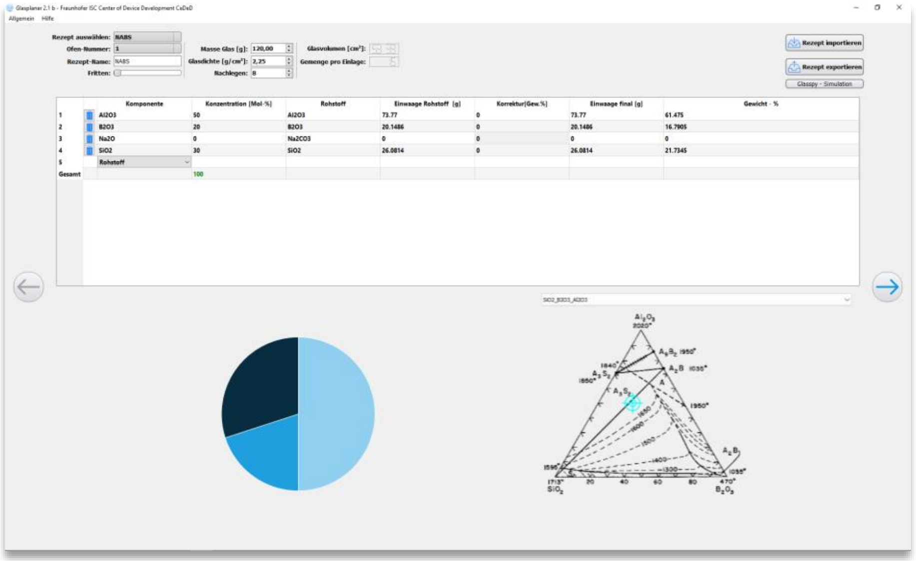
{$P(X_j)$} = **MD** simul glass **properties** LAMMPS of virtual compositions X_j (Δ=5 mol%) {} → ML-modelling (GPR, RF,…) scikit learn PyTorch



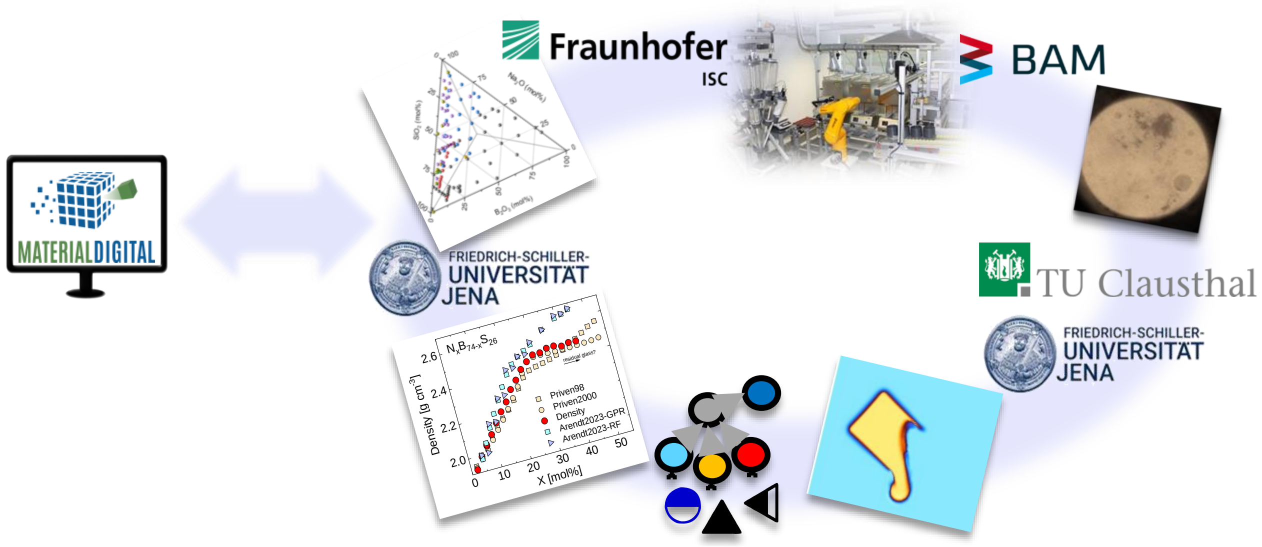
$^+P(C_i) = \rho$, E, G , K, ν, and DFT and U_{ion} *Atomic radii and weighs

Data space
Glass design tools

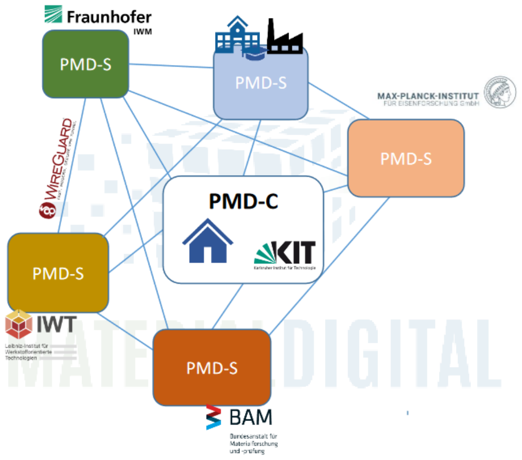




GlassDigital
Digital infrastructure for accelerated glass development



- **Complexity** & **Challenges**, but
- **First steps** regarding all aspects
- **Cooperation** network established
- **After-project cooperation** planned



www.materialdigital.de

Thank you
for your attention

MATERIALDIGITAL

BAM

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Felix Arendt
Marek Sierka

Visibility
Conferences, Workshops, Fairs

2022
07: ICG, Berlin: Talk (M. Kilo), Poster (T. Waurischk)
09: Achema, Frankfurt: Talk (M. Sierka)
09: MSE, Darmstadt: Talk (Y. Chen), Poster (T. Waurischk)
11: MatFo, Köln: Invited talk (R. Müller)
11: Uni Erlangen: Talk (A. Diegeler)
12: UNO IYOG2022, Tokyo, Closing ceremony: Invited talk (L. Wondraczek)

2023
04: OntoCommons Workshop, Berlin: Talk (P. Portella)
05: DGG-USTV, Orleans: Talks (R. Müller (invited), M. Sierka, H. Bornhöft, M. Kilo)
06: GOMD, New Orleans: Invited talk (A. Diegeler)
09: HVG-DGG FA I, Jena: Invited talk (R. Müller)
10: Uni Waterloo, Canada: Talk (A. Diegeler)
11: Istanbul: Sisecam Conf.: Talk (M. Kilo)
11: HVG-DGG Fortbildungskurs, Offenbach: Lectures (R. Müller, M. Kilo, M. Sierka, H. Bornhöft)
12: SIPS Conf, Panama: Talk (A. Diegeler)

2024
04: Analytica, München: Talk (M. Kilo)
05: GTT, Aachen: Talks (T. Waurischk (invited), H. Bornhöft, F. Arendt)
06: Symposium Glasapparatebauer, Mitterteich: Invited talk (T. Waurischk)

Visibility
Paper

BAM

- Pan, Dellith, Wondraczek: Genome Mining in Glass Chemistry Using Linear Component Analysis of Ion Conductivity Data, Adv. Sci. (2023) DOI: 10.1002/advs.202301435
- Arendt, Limbach, Wondraczek, Sierka: Enhancing glass property predictions through ab initio-derived descriptors, JACS (2024) DOI: 10.1111/jace.19904
- Gogula, Bornhöft, Wondraczek, Sierka, Diegeler, Müller, Deubener: Optical Real-Time Castability Evaluation for High-Throughput Glass Melting, Glass Europe (2024) DOI: 10.52825/glass-europe.v2i.1359
- Chen Arendt Bornhöft Camargo Deubener Diegeler Gogula Contreras-Jaimes Kempf Kilo Limbach Müller Niebergall Pan Puppe Reinsch Schottner Stier Waurischk Wondraczek Sierka: Ontology-based digital infrastructure for data-driven glass development, AEM (under review)
- Diegeler Kilo Contreras-Jaimes Waurischk Reinsch Müller: Digital material data-based glass screening for the systematic development of new glasses, Flogen Proceedings (under review)
- Bayerlein et al.: A Unified Concept for a Materials Data Space – Insights from the MaterialDigital Initiative, AEM (under review)
- Schaarschmidt et al.: Scientific Workflows within the Initiative MaterialDigita, AEM (under review)
- 4 others in preparation

- https://github.com/materialdigital/glasdigital-ontology
- https://git.material-digital.de/ya-fanchen/fast-ontodocker

SciGlass Next

BAM

https://sciglass.uni-jena.de/database