NCN Polonez-3
ID Proposal: 351951

SIC-WIN
Reactivity of SI-alloy/C-material systems: Wetting versus INfiltration

Dr Donatella Giuranno
Applicant

Prof Natalia Sobczak
Research Partner

Dr R. Nowak, Eng. G. Burzda, Dr A. Polkowska, Dr A. Kudyba, Dr W. Polkowski
Staff members

Prof Natalia Sobczak

High-T interactions
Metal-Ceramic Interfaces
Joining, High- T Wetting
Experimental Design,
Materials Design,
Composites, etc.

Dr Donatella Giuranno

Metals Surfaces
Thermophys. properties
Wetting characteristics
Fluido-dynamics
Thermodynamics,
Wetting vs Infiltration

Dr Rafal Nowak

Metal-Ceramic wetting
Experimental design,
SEM/Raman
CT by 2D-3D analyses
Cast-alloys, etc.

Dr Wojciech Polkowski

Metal-Ceramics interactions
Experimental design,
LHPC mat., HEA alloys,
React Si/BN, Si/SiC,
T-Mech. Properties,etc.

Dr Artur Kudyba

Metal/Metal Wetting
Metal/C-mat. Wetting
Experimental Design
Apparatuses Design
Lead free solder alloys, etc.

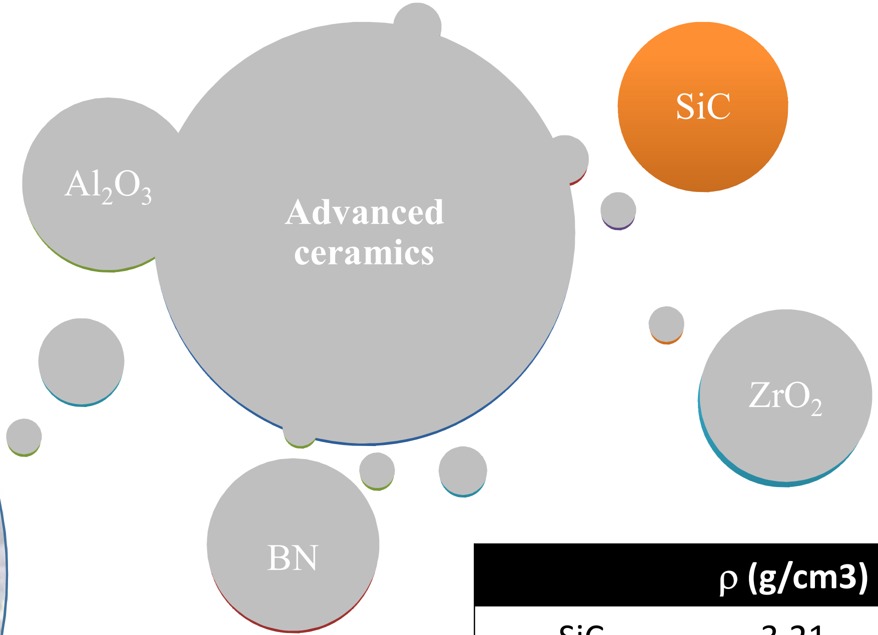
Dr Grzegorz Burzda

Wetting at high T
Mechanical, thermal
funtional properties
Materials characterization
Sample preparation

Dr Adelajda Polkowska

Metal-Ceramic interfaces
Surface charact, HR-SEM
CT by 2D-3D analyses
etc.

Silicon Carbide, a material from another world

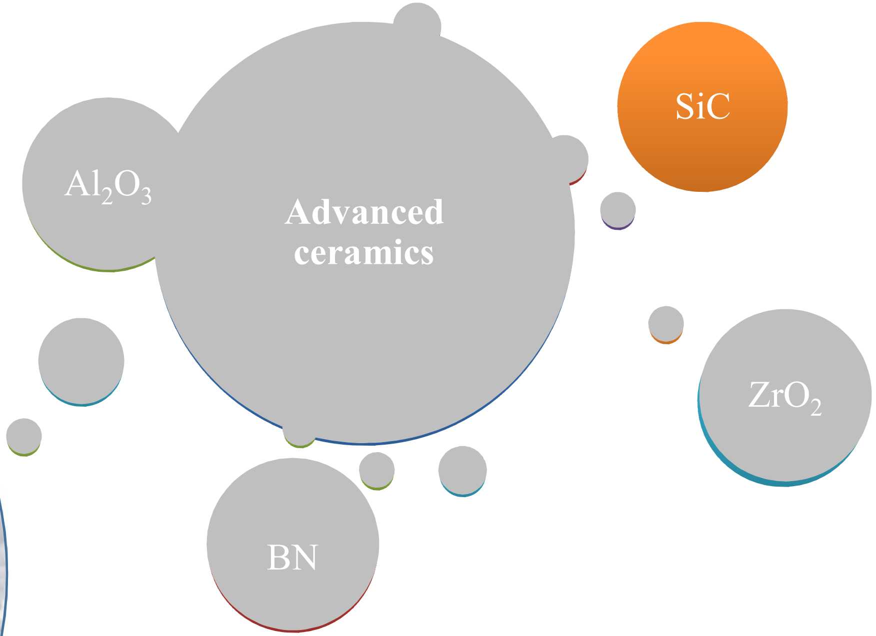


Winning Properties

- ❑ High hardness
- ❑ High chemical inertness
- ❑ Low density
- ❑ High thermal consistency
- ❑ Very good resistance at high temperatures
- ❑ Highly resistant to thermal shocks
- ❑ Good electrical conductivity
- ❑ Is a semiconductor
- ❑ Si and C as alloying additive

	ρ (g/cm3)	E (GPa)	Tm (°C)	K/CTE (10^6 W/m)
SiC	3.21	410	2830	16.5 (α)/16.8(β)
ZrC	6.73	346	3300	3.18
Si_3N_4	3.19	200	1850	12.0
BN (hex)	2.20	30	2700	3.57
Al_2O_3	3.69	300	2050	2.21
ZrO_2	5.68	21.2	2700	0.20
Steel	7.82	200	1500	3.6
Inconel	8.42	157	1370	1.2

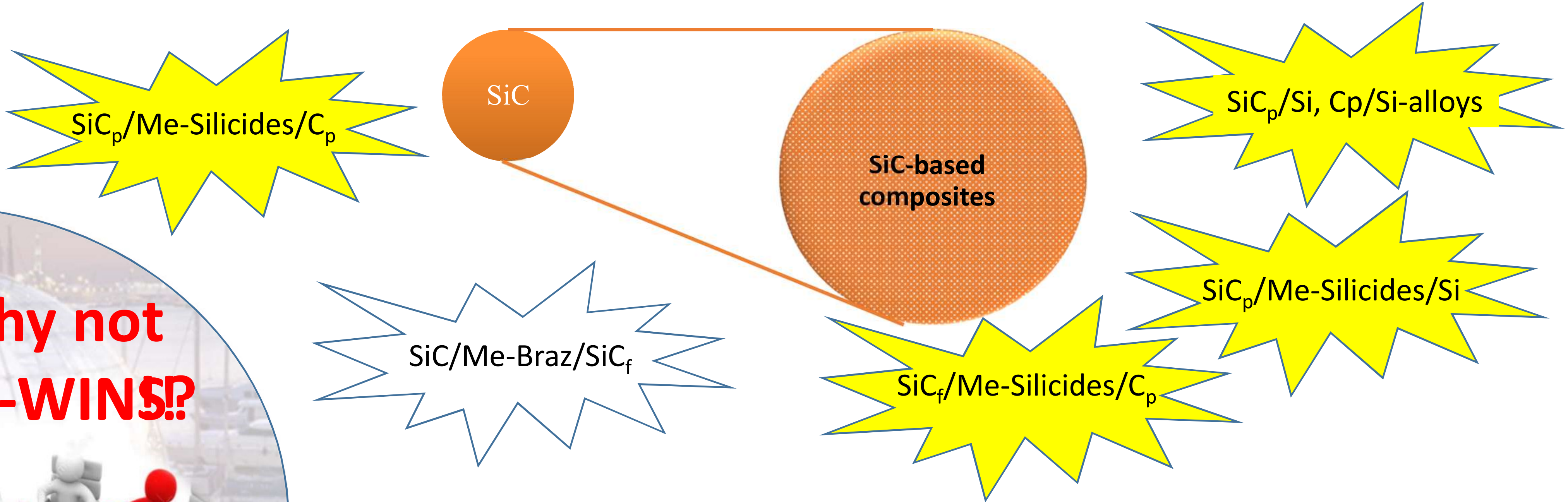
Silicon Carbide, a material from another world

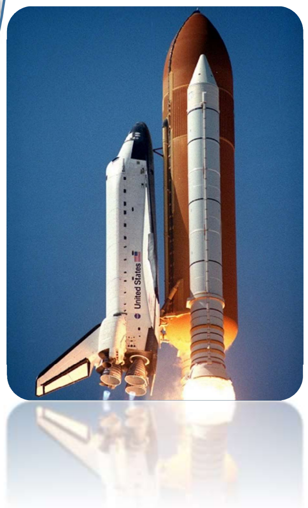


Properties

- ✓ High chemical inertness
- ✓ High temperature resistance
- ✓ Good mechanical properties
- ✓ High thermal conductivity
- ✓ Low density
- ✓ Suitable electrical properties

<u>Powder:</u> Abrasive, steel and MMC-composite reinforcement
<u>Fibers, whiskers:</u> CMC-composite reinforcement
<u>Single crystals:</u> Thyristors (high temperature semiconductor)
<u>Pieces:</u> Resistance, heat exchangers, long-life coatings, nozzles, crucibles, etc





Donatella Giuranno Jan 5th, 2018 NCN Polonez-3 ID proposal: 351951

SiC

SiC-based composites

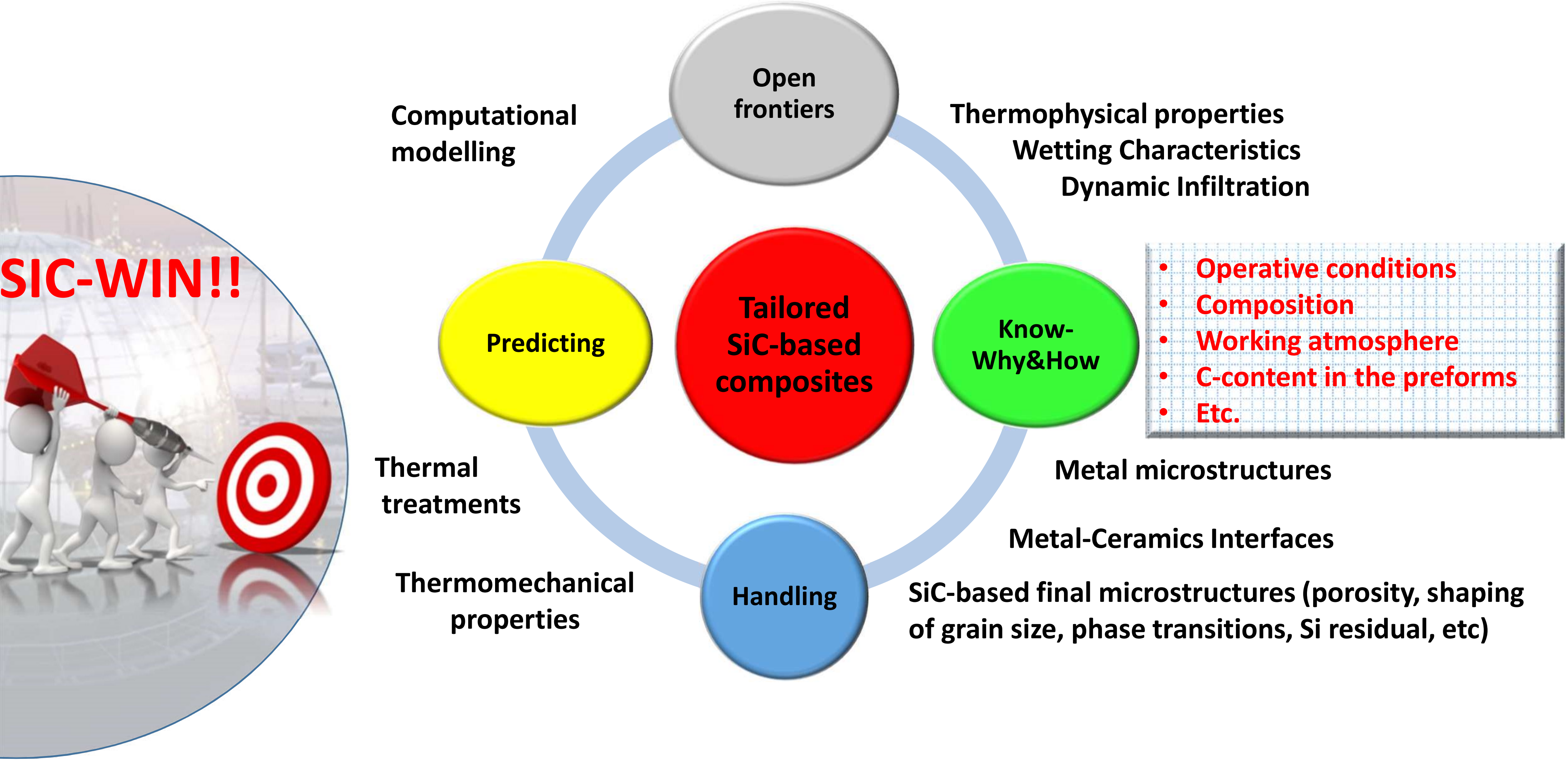
SiC_p/Me-Silicides/C_p

SiC_p/Si, Cp/Si-alloys

SiC_p/Me-Silicides/Si

SiC/Me-Braz/SiC_f

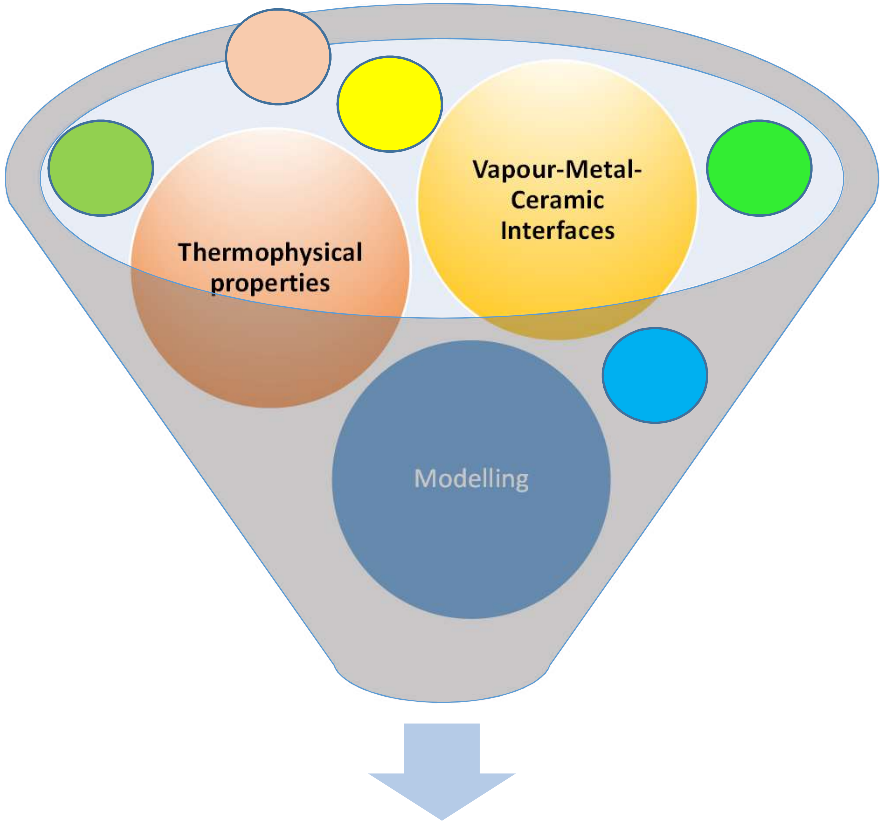
SiC_f/Me-Silicides/C_p



SIC-<u>Systematically</u> WIN!!

DG1

Something of Knew



SIC-WIN: nothing of new!!

Something of New

Diapositiva 8

DG1 Donatella Giuranno; 19/04/2017

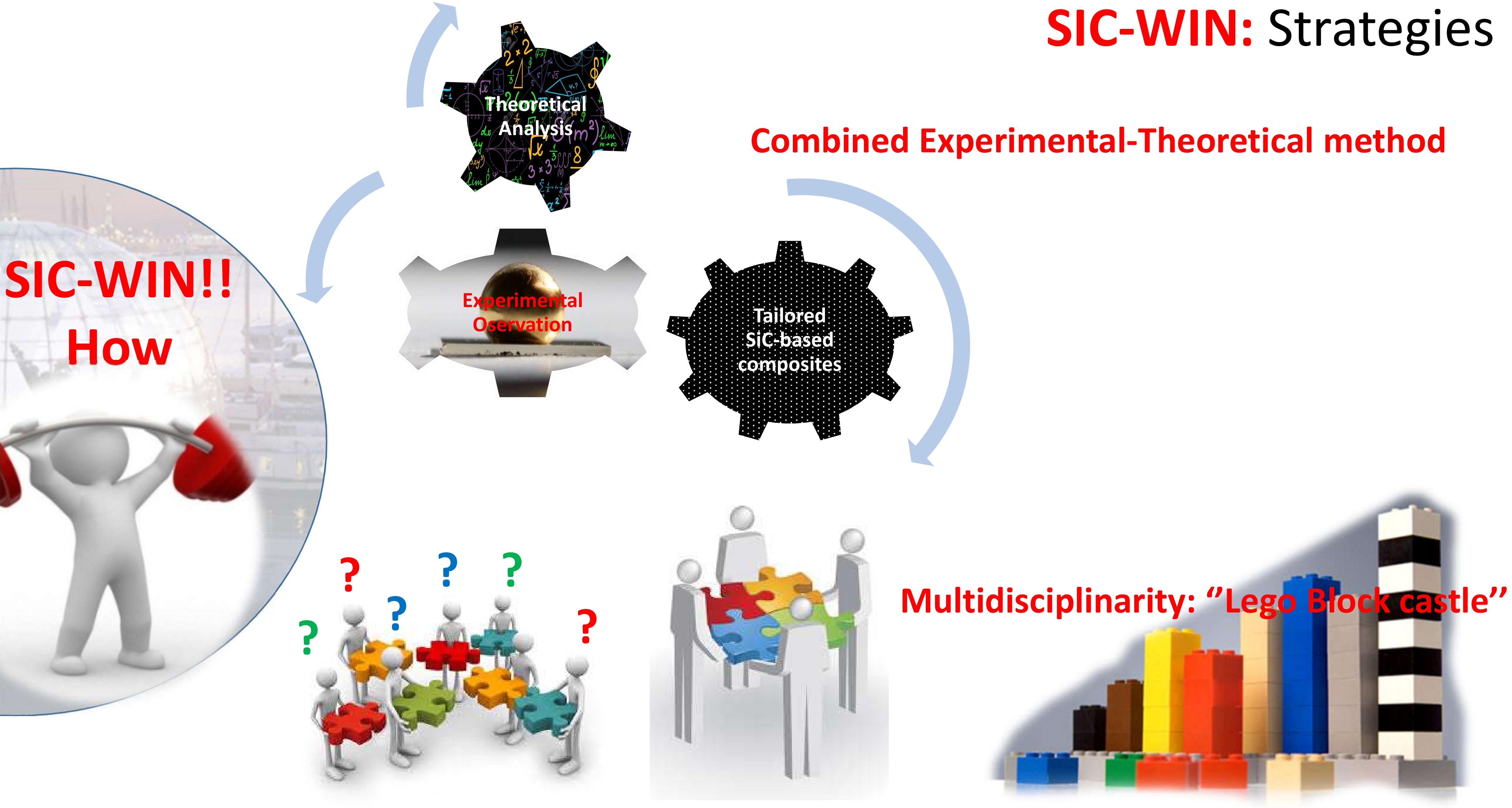
SIC-WIN: Mentality

Material Science: "Closed loop" strategy for Optimization

Processing → Structure → Properties → Performance Application

Material Design: Application driven for Predicting

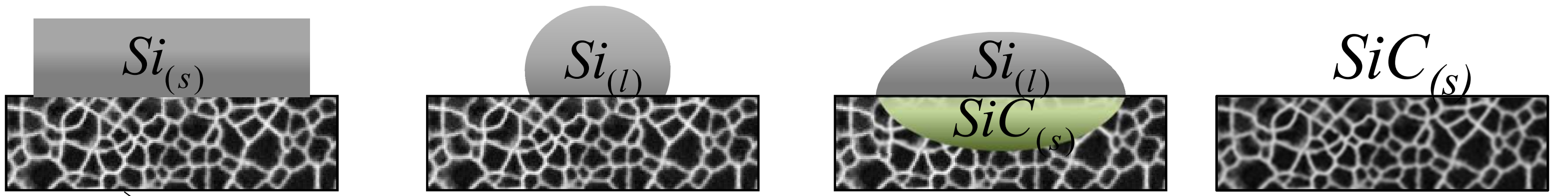
Performance Application → Properties → Structure → Processing


SIC-WIN: Strategies
Combined Experimental-Theoretical method
Theoretical Analysis
Experimental Oservation
Tailored SiC-based composites
SIC-WIN!!
How
Multidisciplinarity: "Lego Block castle"

Reactive infiltration

$$Si_{(l)} + C_{(s)} \leftrightarrow SiC_{(s)}$$

~~T =1900 – 2200 ºC~~
~~P = 10 - 40 MPa~~
~~Y_2O_3, MgO, Al_2O_3~~



C porous → **Reaction Formed Silicon Carbide**

C porous + α-SiC → **Reaction Bonded Silicon Carbide**

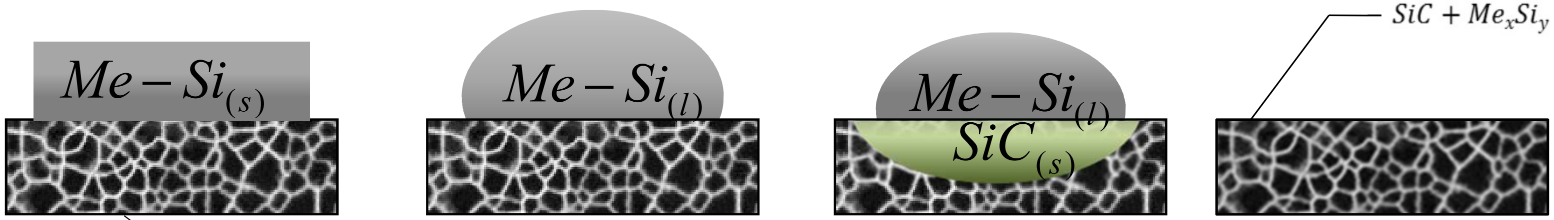
Near-net shape
T =1450 – 1600 ºC
Under vacuum or inert atmosphere

Unreacted carbon
Residual silicon (10-40%)

Reactive infiltration with Si-Me alloys

$$Si - Me_{(l)} + C_{(s)} \leftrightarrow SiC_{(s)} + Me_xSi_y$$

T =1900 – 2200 ºC
P = 10 - 40 MPa
Y_2O_3, MgO, Al_2O_3



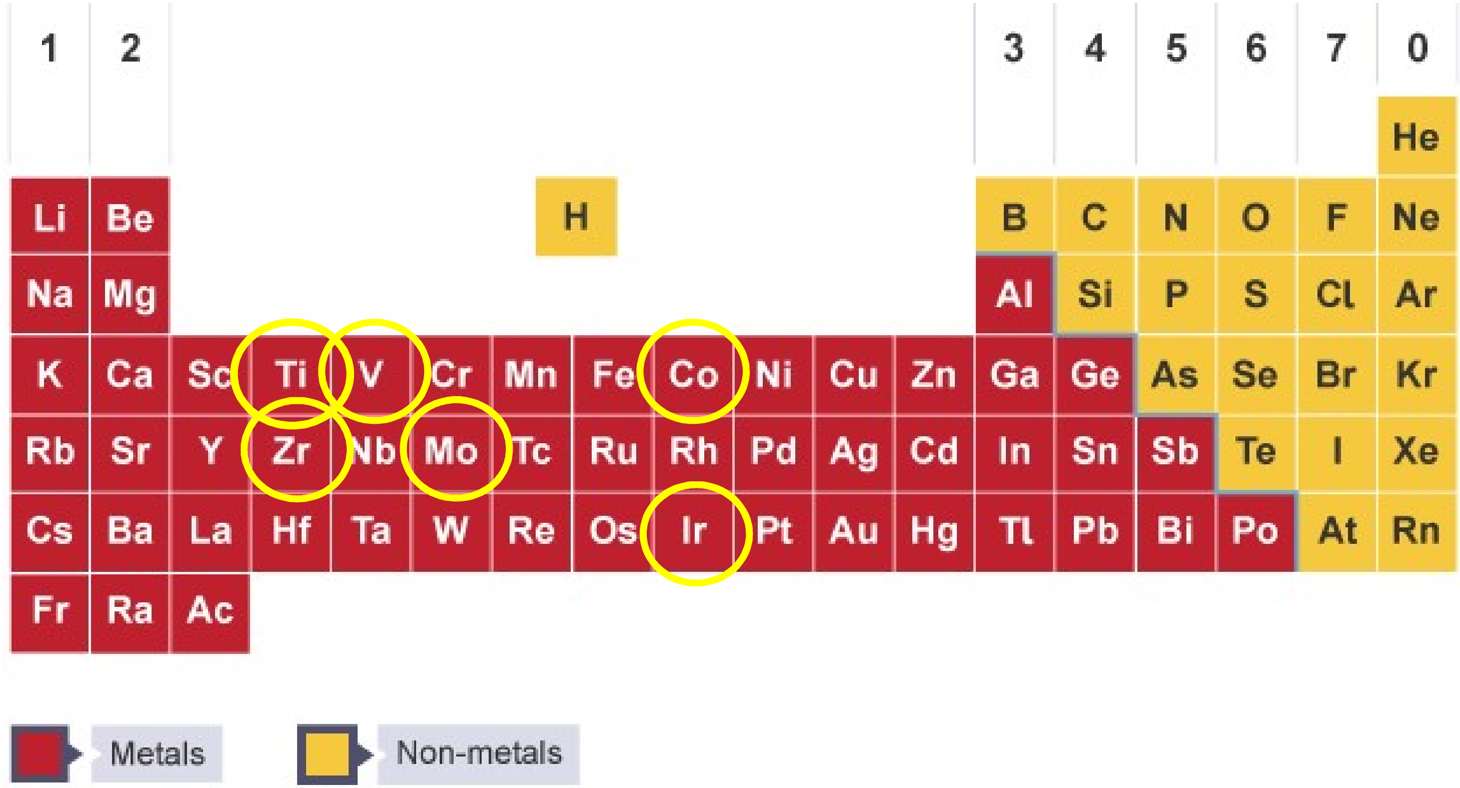
C porous → **Reaction Formed Silicon Carbide**

C porous + α-SiC → **Reaction Bonded Silicon Carbide**

Near-net shape
T =1450 – 1600 ºC
Under vacuum or inert atmosphere

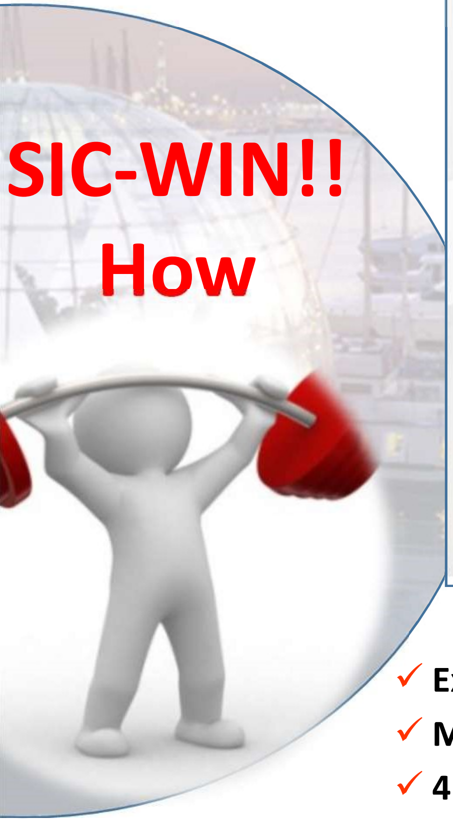
Unreacted carbon
Residual silicon
Reduced

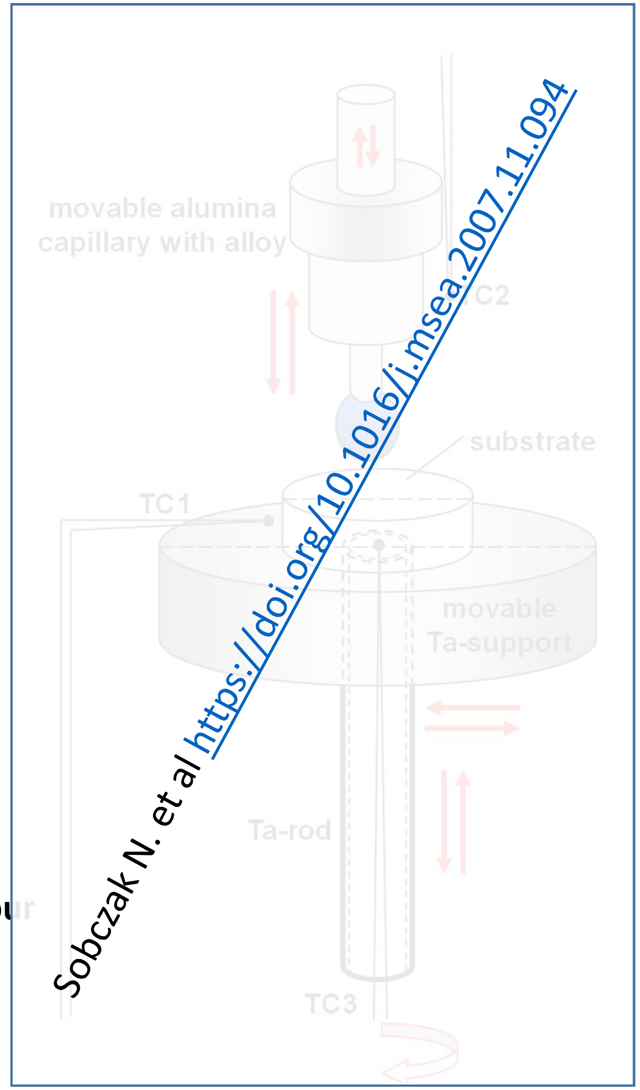
More performance!!!



Type	**Reactivity**	**Metal**
1	No reaction	Au, Ag, Sn, Pb, Ge
2	Me + SiC/C → silicide/C	Ni, Fe, Cu, **Co**, **Ir**
3	Me + SiC/C → Si/Carbide	**V**, Al, Nb
4	Me + SiC/C → silicide/Carbide	**V**, **Zr**, Hf, Cr, Ta, W, **Ti**, **Mo**

Instytut Odlewnictwa
Foundry Research Institute





- ✓ **Experimental temperature: up to 2100ºC, Vacuum down to 10^{-7} mbar (12 pu**
- ✓ **Manipulators for movement of samples, capillary and support**
- ✓ **4 thermocouples for temperature control**
- ✓ **Real-time residual gas analysis**
- ✓ **Methods Sessile +Pendant+Dispensed, etc: Real time acquistion data**

SIC-WIN!! How

Sobczak N. et al https://doi.org/10.1016/j.msea.2007.11.094

- ✓ **Experimental temperature: up to 2100ºC, Vacuum down to 10^{-7} mbar (12 pur**
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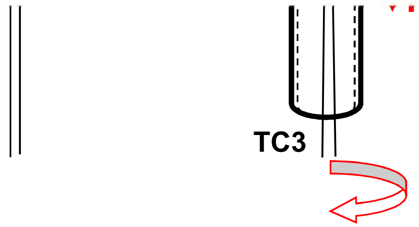

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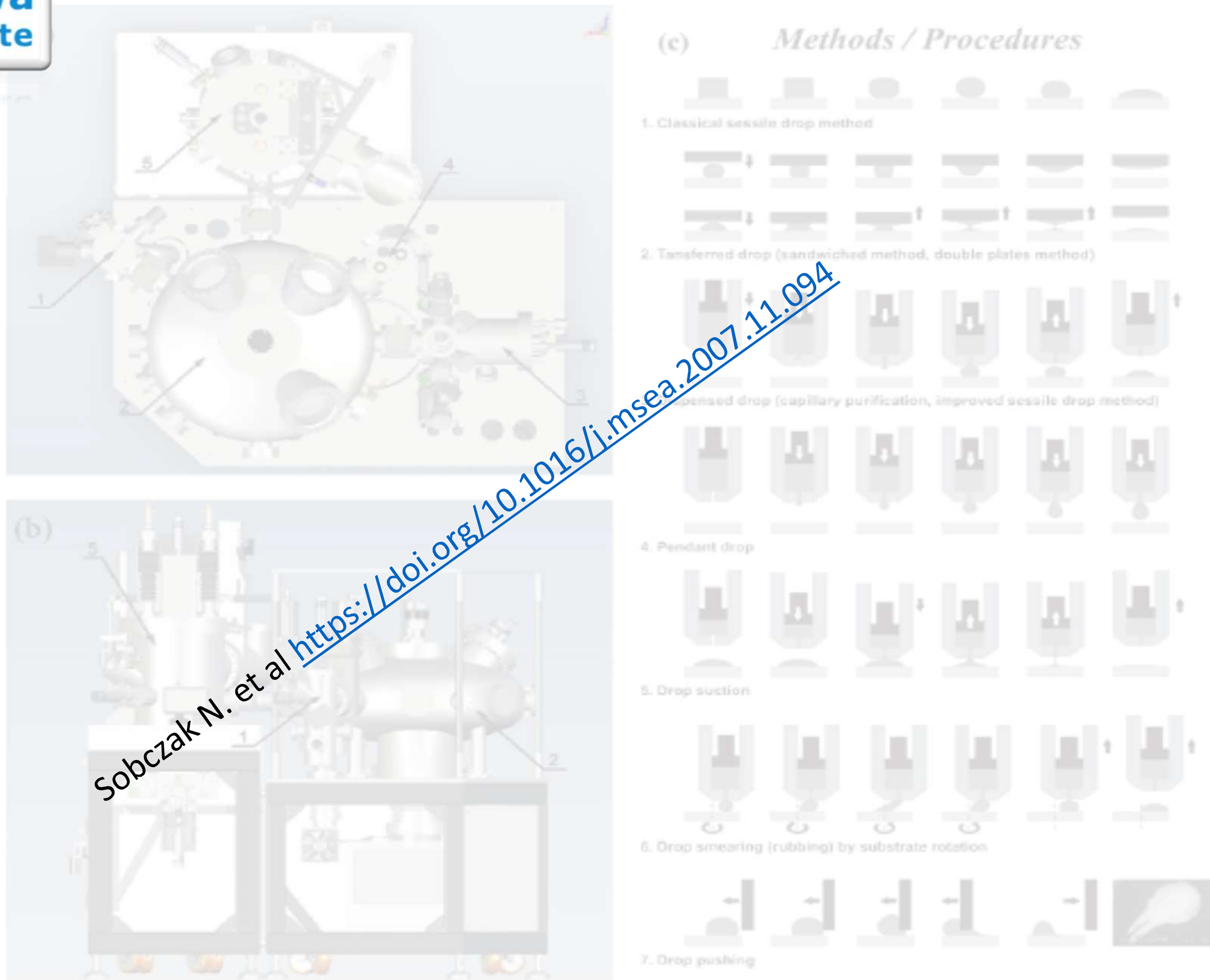
SIC-WIN!!
How



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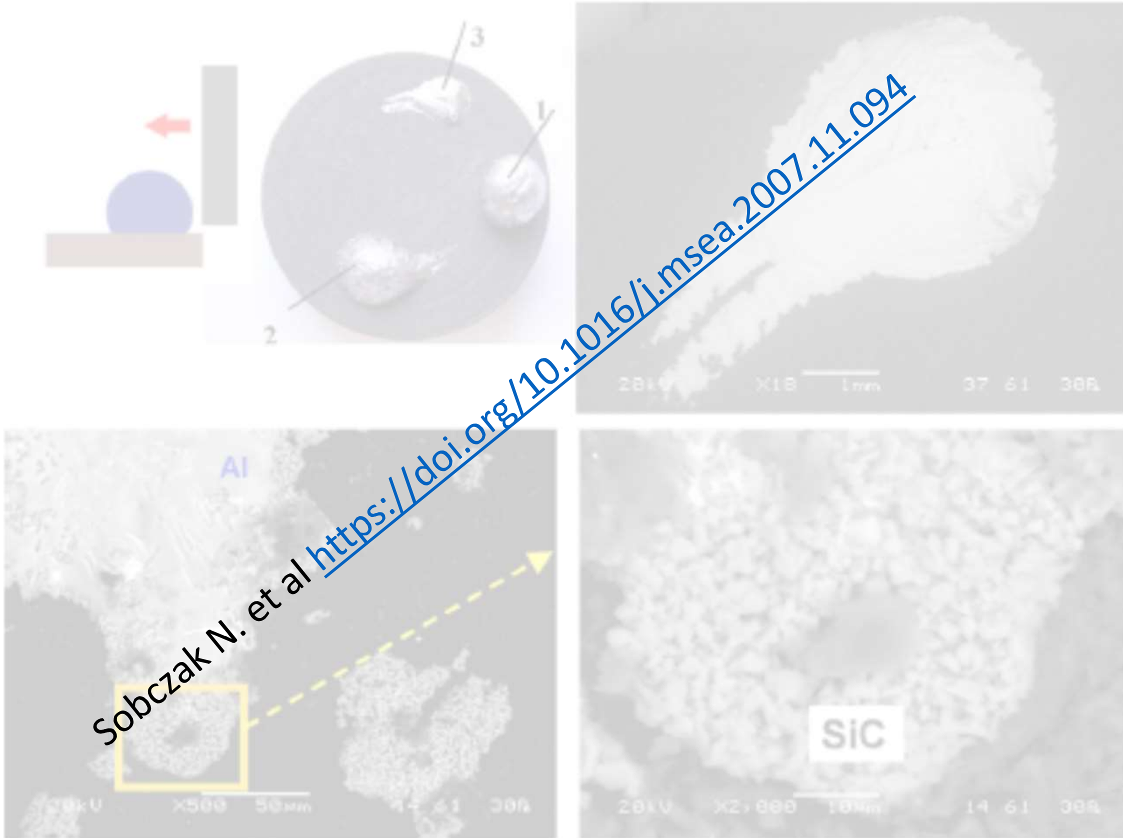


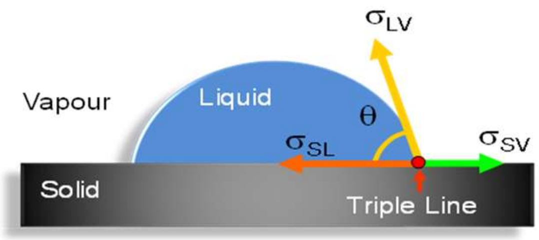
Sobczak N. et al https://doi.org/10.1016/j.msea.2007.11.094

SIC-WIN: Reactivity of SI-alloy/C-material systems: Wetting

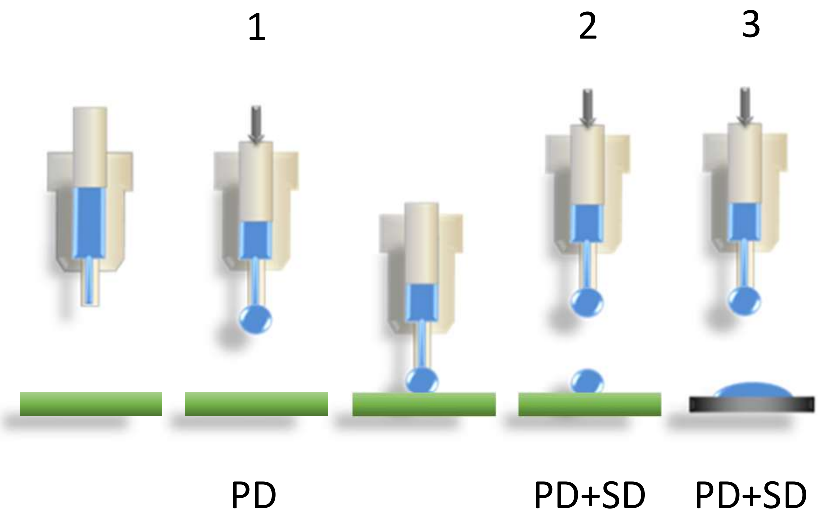
- Dispensed drop technique: well defined operative conditions

SIC-WIN: Surface tension

(& other Thermodynamic and thermophysical properties)



$\sigma_{LV} = f(T, P_{O2}, X_{Si})$



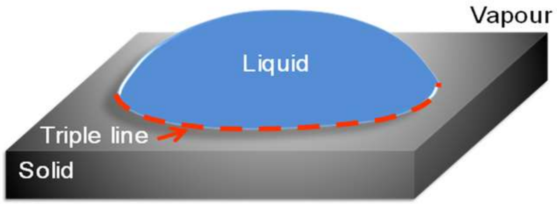
Outcome	1	2	3
σ_{LV}	😐	😐	😐
θ			😐

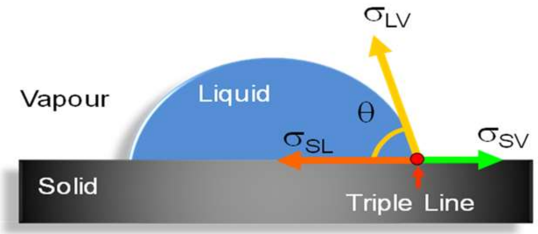
2.....Reproducibility 👍

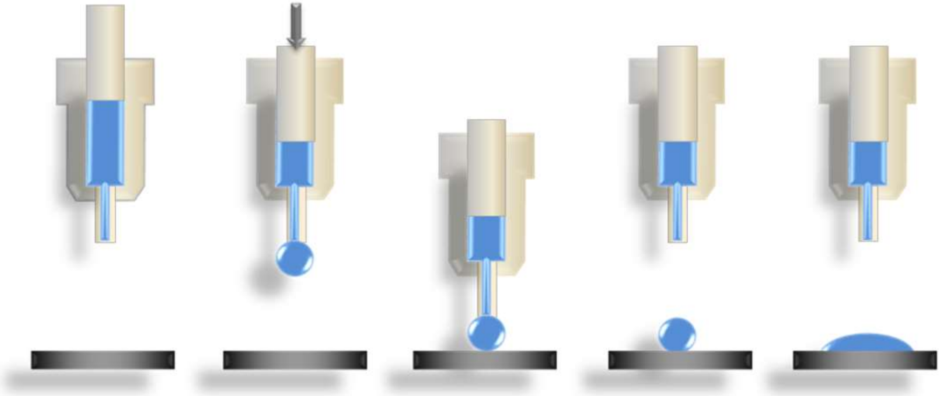
3.....Saving Time 👍 👍

SIC-WIN: Reactivity of SI-alloy/C-material systems: Wetting

- Dispensed drop technique: well defined operative conditions







Wetting: contact angle and interface

Kinetics of spreading [θ(t), 2R(t)],
Ea (T), Reaction products (T), etc.

Interface: SiC crystal growth mechanism

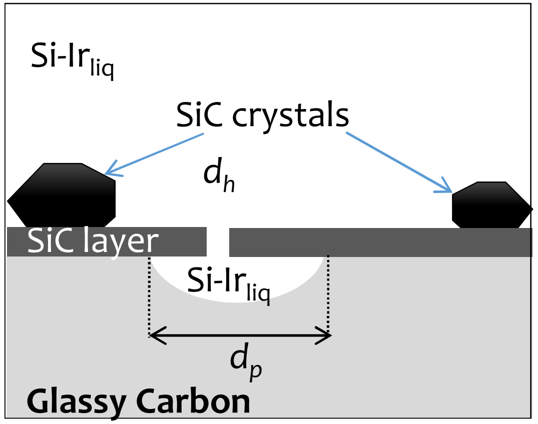
a) Carbon dissolution into liquid: **C(s) → C**

→ formation of the "pockets"

b) C dissolved in the pockets diffuses through the "hole" and the following reaction starts:

$$\mathbf{C + Si_{(liq)} \rightarrow SiC}$$

- equiaxed SiC micron-thick layer: permeable to liquid Si-alloys/solid C still communicates with the outside liquid through pores in the SiC micro-thick layer
- SiC crystals over SiC micron-thick layer
- Packaging of SiC crystals: slower diffusion phenomena



Voytovych et al. J Eur Cer Soc 32 (2012) 3825

The thickness of SiC layer is determined by:

$$e^2(t) = 16 D_c^{liq} \frac{V_m^{SiC}}{V_m^{liq}} (x_1 - x_2) \left(\frac{d_h}{d_p}\right)^2 t$$

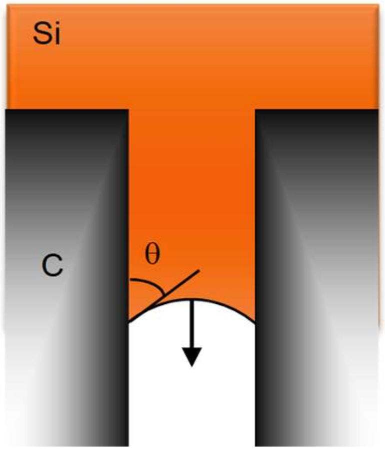
C in liquid Si

Molar volume of SiC e Si

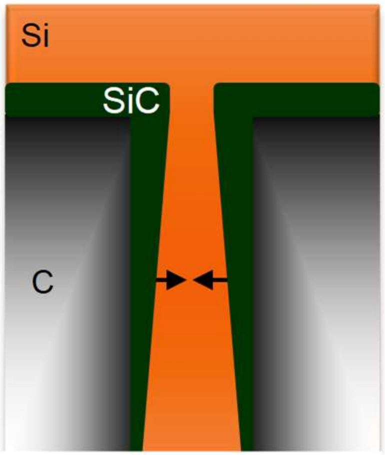
Molar fraction of C in liquid Si in eq with SiC e GC

SIC-WIN: Reactivity of SI-alloy/C-material systems: INfiltration

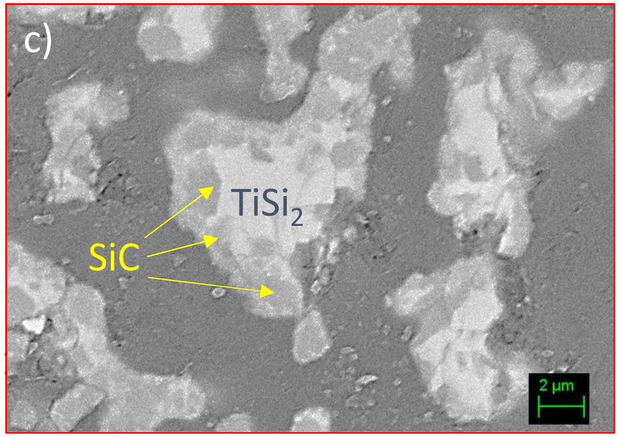
- Darcy's law $h=\frac{2\cdot k\cdot \Delta P}{\eta\cdot(1-V_p)}\cdot t^{1/2}$ Washburn's equation $h^2 = r_{\mathrm{eff}}\frac{\sigma\cos\theta}{2\eta}t$
  - Not reactive system:
    → Good agreement model and experimental values
  - Reactive systems:
    → A large discrepancy (pore closure)



1) Spontaneous infiltration controls the viscous flow



2) Reaction occurs behind the front of infiltration



Infiltrated area
(Si-Ti/C-porous graphite)

SIC-WIN: Reactivity of SI-alloy/C-material systems: INfiltration

Literature survey
-to get theoretical knowledge
-to exploit previous experiences and know-how
-to open and use background on new experimental scenarios

Targeted experiments!!!
saving time and materials consuming!!!
Reliable results as outcomes of standardization
PAPERS!!! CONFERENCES!!! PROFITABLE PRODUCTS!!!

SIC-WIN: Work Plan vs Milestones

30 Nov 2018

Theoretical analysis and modelling

Publications &Report

Mechanical, Thermal and Functional properties

Dynamic Infiltration kinetics

- C-preforms characterization (surface analysis and porosity meas.)
- Infiltration experiments
- Surface and interface analyses by SEM/EDX and XPS
- CT analysis by 2D/3D Imaging of SiC-based composite

Wetting characteristics

- Contact angle measurements by dispensed drop
- Spreading kinetics and reactivity studies
- Interface characterization by SEM/EDX and XPS

Thermophysical properties

- Selection of suitable container material
- Surface tension and density measurements by pendant and large drop methods
- Thermodynamic calculation

Processing of raw materials

1 Dec 2017

Literature survey

Critical issues

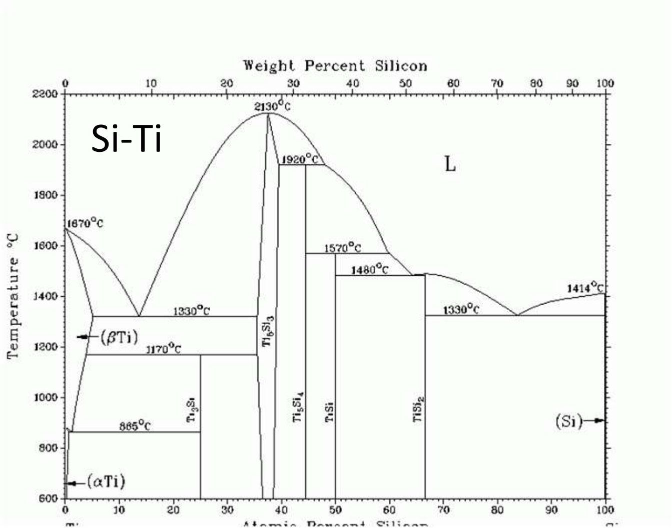
High temperature melts → **High reactivity**

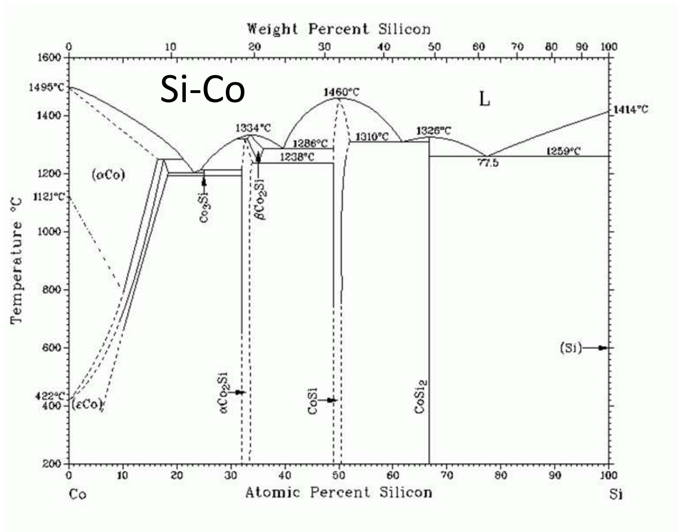
SIC-WIN: Work Plan vs time

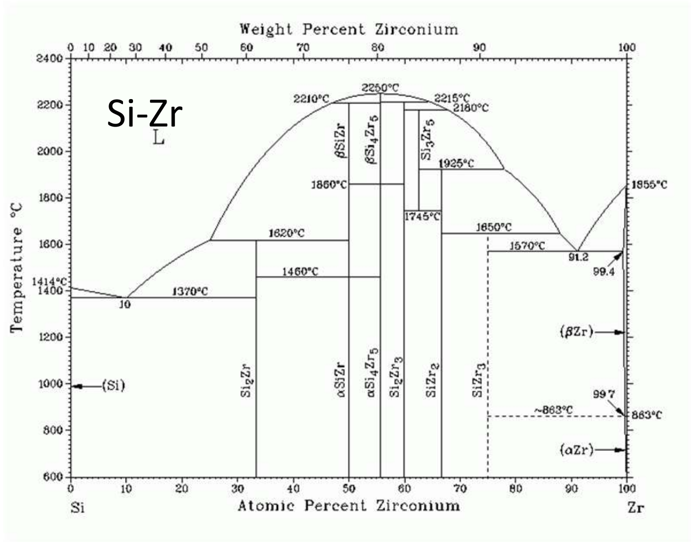
Milestones (outline of the work and critical paths)	1	2	3	4	5	6	7	8	9	10	11	12
Literature survey	■	■	■	■	■	■	■	■	■	■	■	■
Processing of raw materials	■	■										
Thermophysical properties • Selection of suitable container material • Surface tension and density measurements by pendand and large drop methods • Thermodynamic calculation		■	■ 😐	■								
Wetting characteristics • Contact angle measurements by dispensed drop • Spreading kinetics and reactivity studies • Interface characterization by SEM/EDX and XPS				■	■ ☺	■						
Dynamic Infiltration kinetics • C-preforms characterization (surface analysis and porosity measurements) • Infiltration experiments • Surface and interface analyses by SEM/EDX and XPS • CT analysis by 2D/3D Imaging of SiC-based composite						■	■	■ ☺	■			
Mechanical, Thermal and Functional properties								■	■ ☺	■		
Analysis of the results and development of theoretical and Experimental correlations			■	■	■	■	■ 😐	■	■	■	■	
Pubblications and Report				■	■	■	■	■	■ ☺	■	■	■
Each box represents 1 month												

SIC-WIN: Surface tension & density measurements

$\rho_L, \sigma_{LV} = f(T, X_{Si})$ & $f(P_{O2})$



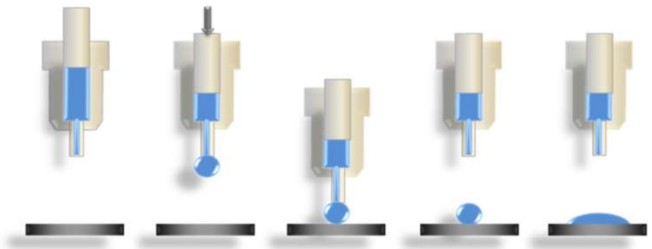




a)

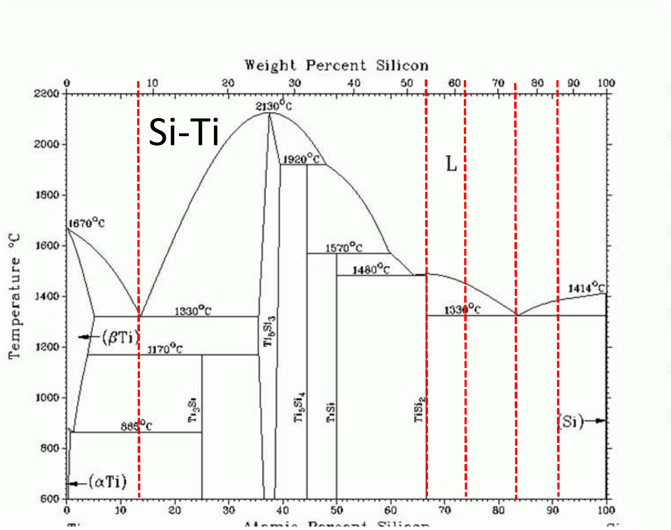
Contact heating vs Capillary purification

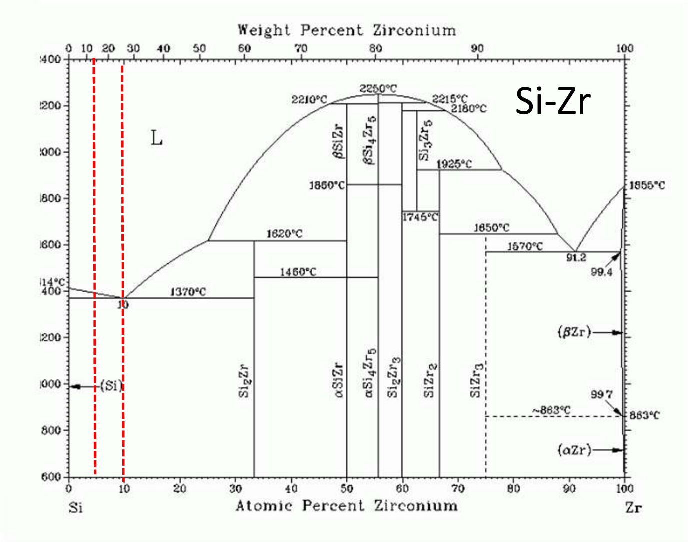
b)



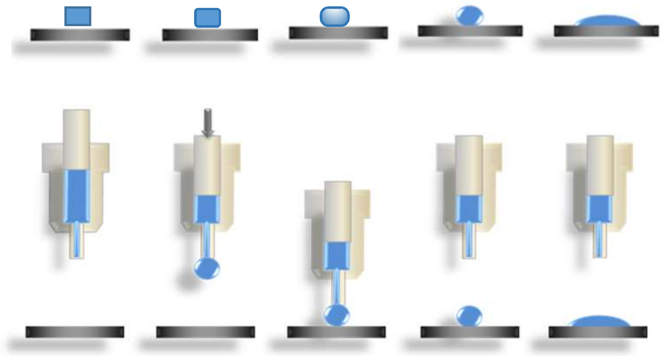
SIC-WIN: Wetting and Interface characterization

$\theta = f(t, T, X_{Si})$ & f (GC, SiC, Graphite, etc)





a)



b)

Contact heating vs Capillary purification

SIC-WON: Expected impacts

SiC-based composites obtained by **RBSC optimized** processes: lower processing temperatures, shorter times and near-net shape fabrication capabilities

Several crucial issues in manufacturing of new/more performant refractory SiC-based composites for highly demanding applications via RBSC process **will be addressed**

Insights on reactive infiltration and knowledge on systems not yet investigated

Reliable surface tension and density data will be obtained, as fruitful inputs data in **computational models** used to **optimize** casting and solidification processes

SIC-WON: Expected benefits for SIC-WIN Team

GREAT opportunity to BOOST the PI's research career further in an enlarged international outstanding scientific environment.

Get access to experiments/investigations to be performed by the HI outstanding and unique facilities and to be open and ready for new and challenging R&D "frontiers".

A significant amount of scientific publications in the fields of advanced metallurgy, ceramics, MMC and CMC composites will be produced resulting in the increase of h-index and a general quotation in the HTC-community

Further consolidation of the already existing ITA-POL collaboration by merging own competencies and to open in a next future new possible common R&D activities for the new forthcoming calls (HORIZON 2020)

The SIC-WIN outcomes and the incoming knowledge/achievements will enable the PI to consolidate her scientific background, for building independently her own research group and to be open, ready and capable in focusing on new research fields.

SIC-WON: Expected benefits for SIC-WIN Team

Further consolidation of the already existing ITA-POL collaboration by merging own competencies and to open in a next future new possible common R&D activities for the new forthcoming calls (HORIZON 2020)

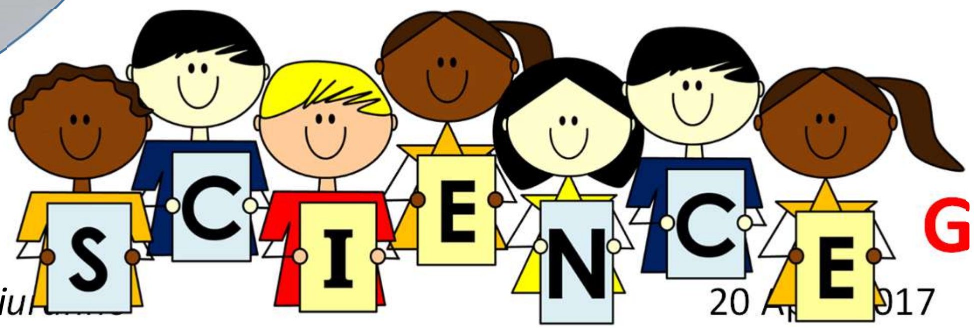
Significant amount of scientific publications within this specific field of metallurgy, ceramics and composites will be produced with an increase of h-index and a general quotation in the HTC-community

NEW knowledge and background as well as new “products” obtained (patented???).

The results and the new knowledge/background will provide excellent basis to the younger team-members for building in a next future their own research group: “new generation” of CHTS with a stronger and consolidated scientific background and know-how.







SIC-WON

Growing up together!!!