Innovative SiC coating protecting graphite reactor components in SiC semiconductor processing

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Motivation

Challenge of material degradation

- Inner reactor parts made of graphite and/or SiC which are used in SiC semiconductor processing are highly stressed by high temperatures and corrosive gas environment
- This results in erosion and strong degradation of the reactor parts leading to high maintenance effort, extended reactor down time and considerable spare part costs
- → High quality SiC coatings on graphite parts could be a promising approach to
- a) effectively protect graphite parts from harsh environments and increase their lifetime
- b) be an economic alternative in comparison to cost intensive SiC full-ceramic parts

Potential Application

SiC epitaxy reactors

• SiC coatings **protect graphite hot zone parts** from corrosion by reactive gases like SH_4 or H_2 at high temperatures up to 1600°C. Moreover, SiC epi layer material quality is improved by avoiding loose particle ablation without any additional contamination.

SiC oxidizers

SiC coated graphite parts withstand oxidizing atmospheres and may be used in oxidizers as a cost-effective alternative to bulk SiC material, ensuring a reliable supply chain due to readily available raw materials.

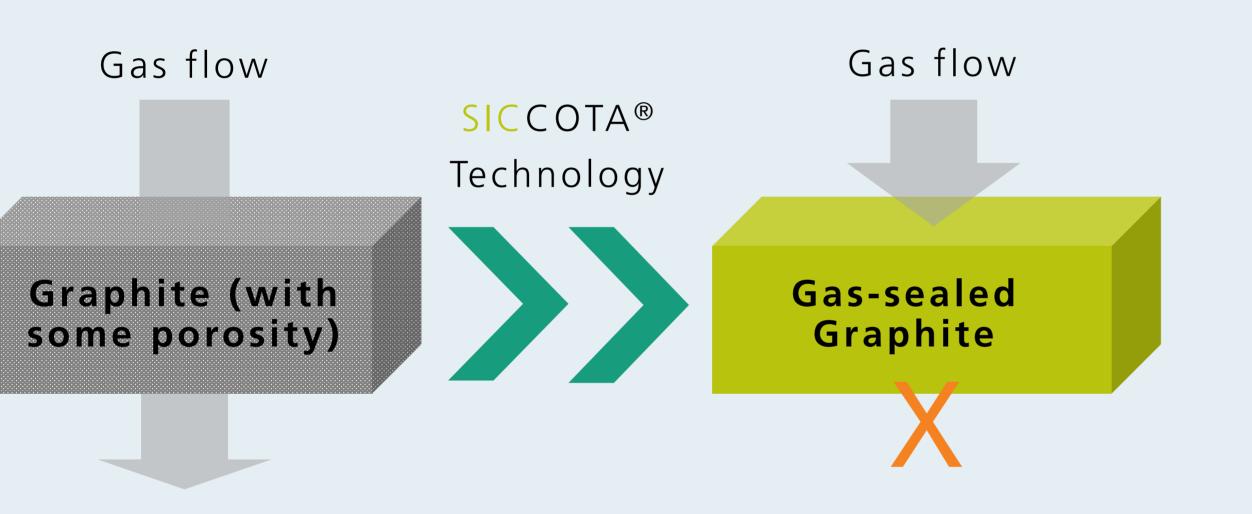
SICCOTA® coating technology and characteristics

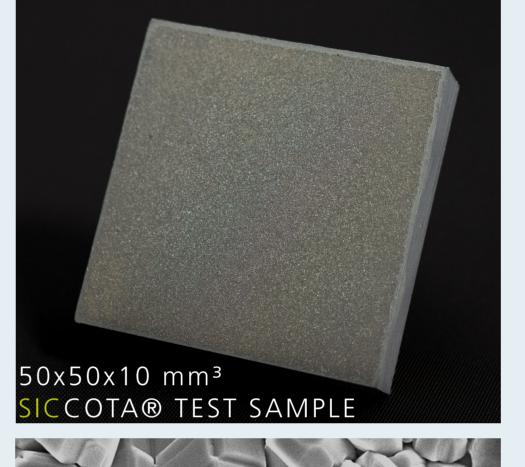
Aim

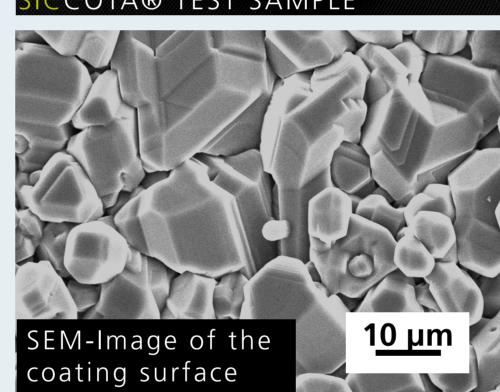
 Reducing stress and avoiding degradation of graphite parts caused by high temperatures and reducing/ oxidizing environments during semiconductor processing

Approach

Sealing porous graphite by converting its surface into SiC



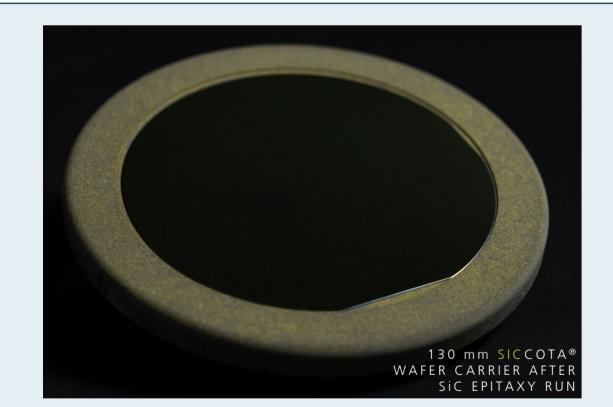




Coating properties

- Polycrystalline SiC consisting of 99.97 % cubic SiC
- Good wear resistance and excellent adhesion to the substrate (> 8 MPa adhesive strength)
- Low gas permeability due to highly dense crystalline
 SiC-structure
- Resistance against corrosive, reducing (e.g. H_2) and oxidizing (e.g. O_2) gases
- Partial and all around coating possible
- → Cost-effective and highly flexible technology

Contamination test in SiC-epitaxy



- Ø100 mm SiC wafer on top of coated wafer carrier
- Growth of ~15 μ m SiC Epi-layer in 1h @ 1600°C under SiH₄, C₃H₈, H₂, N₂ atmosphere
- Measuring surface contamination on SiC wafer surface by after Epi-run

limit (dl)	Species	SiC Wafer		SiC-Wafer	epitaxy
lillit (di)		(Si-plane)		(Si-plane)	process
0.031	Li	<dl< td=""><td></td><td><dl< td=""><td>n.m.</td></dl<></td></dl<>		<dl< td=""><td>n.m.</td></dl<>	n.m.
0.126	Na	7.157		1.068	n.m.
1.125	Al	1.583		<dl< td=""><td>n.m.</td></dl<>	n.m.
0.115	K	11		0.364	0.053
0.738	Ca	2.641		18.78	<dl (0.05)<="" td=""></dl>
0.016	Ti	1.52		<dl< td=""><td><dl (0.03)<="" td=""></dl></td></dl<>	<dl (0.03)<="" td=""></dl>
0.063	V	1.58		<dl< td=""><td><dl (0.02)<="" td=""></dl></td></dl<>	<dl (0.02)<="" td=""></dl>
0.471	Cr	<dl< td=""><td></td><td><dl< td=""><td>0.105</td></dl<></td></dl<>		<dl< td=""><td>0.105</td></dl<>	0.105
0.051	Mn	<dl< td=""><td></td><td><dl< td=""><td>0.488</td></dl<></td></dl<>		<dl< td=""><td>0.488</td></dl<>	0.488
0.270	Fe	0.385		<dl< td=""><td>0.306</td></dl<>	0.306
0.108	Со	0.412		<dl< td=""><td><dl (0.02)<="" td=""></dl></td></dl<>	<dl (0.02)<="" td=""></dl>
0.363	Ni	1.448		<dl< td=""><td><dl (0.25)<="" td=""></dl></td></dl<>	<dl (0.25)<="" td=""></dl>
0.618	Cu	<dl< td=""><td></td><td><dl< td=""><td><dl (0.14)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.14)<="" td=""></dl></td></dl<>	<dl (0.14)<="" td=""></dl>
0.032	Zn	0.3		<dl< td=""><td><dl (0.05)<="" td=""></dl></td></dl<>	<dl (0.05)<="" td=""></dl>
0.565	As	<dl< td=""><td></td><td><dl< td=""><td><dl (0.21)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.21)<="" td=""></dl></td></dl<>	<dl (0.21)<="" td=""></dl>
0.015	Sr	<dl< td=""><td></td><td><dl< td=""><td><dl (0.003)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.003)<="" td=""></dl></td></dl<>	<dl (0.003)<="" td=""></dl>
0.008	Cd	<dl< td=""><td></td><td><dl< td=""><td><dl (0.004)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.004)<="" td=""></dl></td></dl<>	<dl (0.004)<="" td=""></dl>
0.010	Ва	0.012		0.051	<dl (0.001)<="" td=""></dl>
0.001	Та	0.077		0.066	0.058
0.012	Pb	<dl< td=""><td></td><td><dl< td=""><td><dl (0.002)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.002)<="" td=""></dl></td></dl<>	<dl (0.002)<="" td=""></dl>
0.002	Bi	<dl< td=""><td></td><td><dl< td=""><td><dl (0.001)<="" td=""></dl></td></dl<></td></dl<>		<dl< td=""><td><dl (0.001)<="" td=""></dl></td></dl<>	<dl (0.001)<="" td=""></dl>
Values for surface contamination in 1E10 at/sm2					

Processed Unprocessed Industrial

Values for surface contamination in 1E10 at/cm², measured by VPD ICP-MS

Results

- No ablation or cracking of the SiCCOTA® coating after several epitaxy processes
- No significant metal contamination measured by VPD ICP-MS
- No influence of graphite components on contamination behaviour due to complete isolation by the SiC coating from growing atmosphere

Oxidation resistance experiments

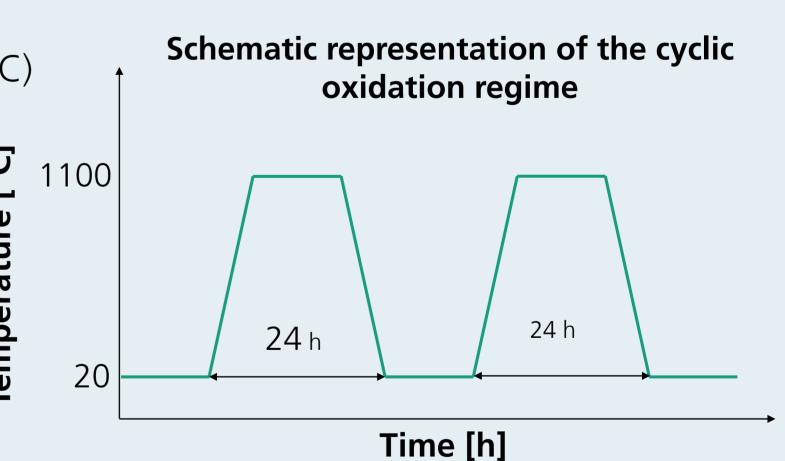
Cyclic oxidation (24 h intervals @ 1100°C)

Heating rate of 200 K/min

Continuous air flow (20% O₂)

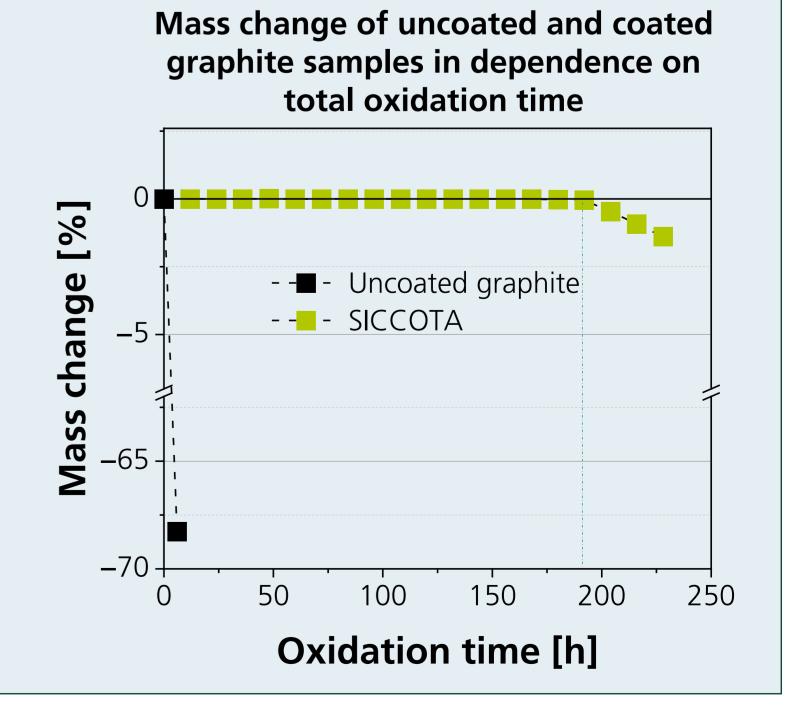
Sample size: 50 x 25 x 10 mm³

Measuring the mass loss of coated samples after each cycle



Results

- Uncoated graphite sample (reference)
 with massive weight loss already within
 1st cycle
- In case of SiCCOTA® sample an oxidation resistance (equals to mass loss < 0.1 %) of up to 182 hours could be demonstrated
- SiC coating perfectly withstands cyclic temperature changes due to excellent adhesion to the substrate



Conclusion

SiCCOTA®

- Represents a new cost-effective technology for protection of graphite parts vs. extremely high temperatures and corrosive gas atmospheres like silane, hydrogen or oxygen
- Shows no ablation or crack-formation and no significant metal contamination

SiCCOTA®

is a promising alternative to other SiC coating approaches (like CVD) and/or SiC bulk material used in SiC semiconductor processing