

Ceramic Product Test

ECOMONDO 2011

Agenda

- Introduction
- The company LiqTech International A/S
- Goals of "Ceramic Product Test" WP5
- Production of test samples (sintering)
- Test results
- Conclusions



Introduction

 LiqTech is an end-user of SiC powder and role as test partner



Waste tyres

Silicon Carbide



LiqTech International A/S

- LiqTech International A/S Liquid Technology International A/S
- Formerly known as CoMeTas
- Development, production and marketing of:
 - SiC membranes
 - Catalytic coatings for the automotive industry
 - Kiln furniture
 - Founded in February 2006
 - Located in Ballerup , West of Copenhagen, Denmark
 - Production of 2.500 m² p.a.
 - Patent rights for SiC membranes





Products

Diesel particle filters



Ceramic membranes



Kiln furniture







Unique membranes

- Highest flux
- Total chemical resistance
- Temperature resistance
- 100 % Silicon Carbide





LiqTech makes a difference



Produced water



Pre-filtration for reverse osmosis







Waste water treatment



Drinking water







SEM picture of 2 layer membrane

- SiC content: > 97%
- Full chemical resistance:
 0 to 14 pH value
- High thermal robustness
 - Maximum application temperature: 1000°C
- Porosity: 45%
- Thermal and electric conductive
- Highly hydrofilic
- Environmental safe





WP5 goals

- To show that TyGRe SiC powder can be used for industrial processes and hereby can be sold as a commercial product
- To compare the TyGRe powder to an existing product (of commercial SiC)



Demands for the TyGRe SiC

- High content of α-SiC
- 2. Pure SiC powder
 - Prefered above 95%
- 3. Narrow particle size distribution
- 4. Small size grains



Result of produced TyGRe SiC powder



Production of SiC powder

	SiC %	SiO2 %	Si %	Fe %	Al %	Free C %
SIKA I						
(Refractories)	99,7 (97)	0,15	0,02	0,02	0,02	0,15
SIKA II						
(Refractories)	99,5 (94-97)	0,2	0,02	0,04	0,03	0,15
SIKA III						
(Refractories)	99,2 (90-94)	0,35	0,02	0,5	0,25	0,15
SIKA IV						
(Refractories)	99 (>90)	0,45	0,05	0,8	0,3	0,2

• Acheson





Production of SiC powder

- Two production methods of powder:
 - Batch tubular furnace ENEA
 - Continuous plasma process IMEC



Production of test samples



- Two types of samples
 - Discs
 - Pellets
- Forming of pellets are performed during liquid phase and SPS sintering





Production of test samples



Forming

• Weighing

Pressing



Sintering parameters

- SiC powder
- Sintering aids
- Sintering temperature
- Sintering pressure
- Residence time
- Three types of sintering:
 - Vapour phase sintering LiqTech
 - SPS (Spark Plasma Sintering) IMEC
 - Liquid phase sintering ENEA





Vapour phase sintering

- The transport path during vapour phase sintering
- a) grain boundary diffusion.
- b) lattice diffusion
- c) evaporationcondensation
- d) surface diffusion





Spark plasma sintering (SPS)





- Process proceeds through three stages:
 - 1. Plasma heating
 - 2. Joule heating
 - 3. Plastic deformation



Liquid phase sintering



- Stages of liquid sintering
- a) Liquid phase
- b) Solution-precipitation
- c) Coalescence



Test methods of samples

SEM		EDX		Density
 Visual evaluation of sintering 		 Identification of pollutions after sintering 		 Identification of material physical property

- SEM Scanning Electronic Microscope
- EDX Energy Dispersive X-ray spectroscopy



SEM

• Pellet (LPS)



• Disc (SPS)





EDX





Density

- At last deliverable sample E had the highest density: 89.2%
- The theoretical densities used are
 - 3.2 g/cm3 for vapour phase sintering and
 - 3.23 g/cm3 for liquid phase sintering





Test results

- Best result to date
 - Sintered pellet
 - SiC powder produced by thermal plasma synthesis by IMEC
 - Liquid phase sintering by ENEA
 - Sintered density 91.3% T.D. corresponding to 2.95 g/cm3





Benchmark of powder

- Different purity
 - TyGRe powder has a higher content of metallic impurities than commercial powder
- Density
 - TyGRe powder has the density of 91% T.D. (sample obtained by means of liquid phase sintering)



Conclusions

- Impurities in SiC powder cause poor forming and sintering properties
 - Impurities are created in the production of SiC powder
- Correct purification methods are very important
- Relevant part of the research work in WP5 was to find a valid procedure to purify the raw TyGRe SiC

