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# ***Beryllium Qualification Activity for ITER First Wall Applications***

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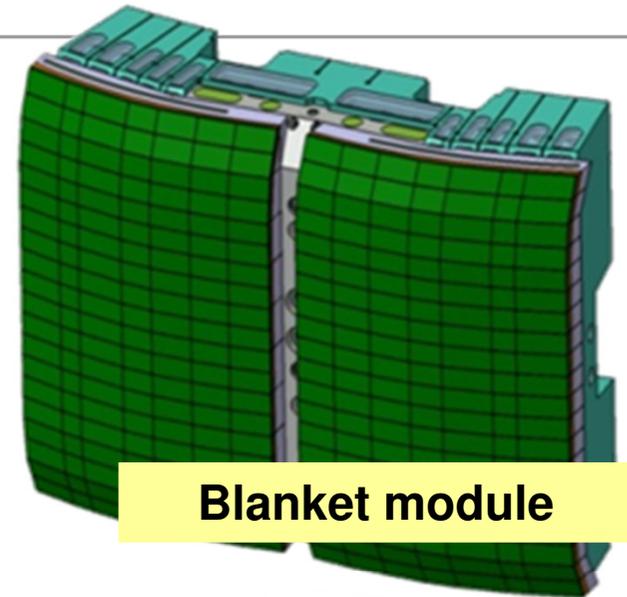
# Outline

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- Introduction
- Selection of Beryllium grades
- Proposal of new grades
- Qualification program for RF and CN beryllium
  - Manufacturing technology of RF and CN grades
  - Main thermal and mechanical properties
  - Behavior at thermal shock/VDE/cyclic tests
- Summary

# Introduction

- Beryllium is considered as armour material for the ITER First Wall since beginning of the ITER EDA phase. Main reasons:
  - Oxygen gettering
  - Low Z, Plasma compatibility

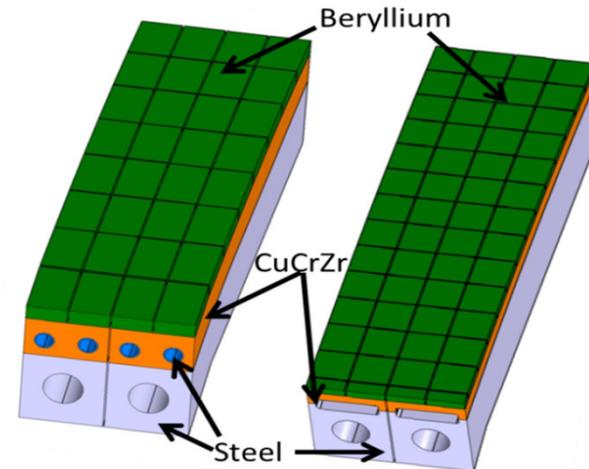


## Current design:

- Thickness of tiles 6-10 mm,
- Various sizes (e.g. 25x25mm, 50x50mm)
- Total net weight - ~10 tons

See talk by T. Hirai

See talk by R. Mitteau



- Normal heat flux panels 1 – 2 MW/m<sup>2</sup>
- Enhanced heat flux panels 3.5 – 5 MW/m<sup>2</sup>

# Introduction

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- During many years of ITER activities, the selection of specific beryllium grade(s) has been under study.
- The selection of the optimum grade is driven by those properties, which are very sensitive to the impurity levels, grain size, methods of production, thermo-mechanical treatment, and which usually differ for the different beryllium grades.
- Depending on the above mentioned parameters, mechanical properties, thermal shock/thermal fatigue resistance, and properties after neutron irradiation are different for different beryllium grades
- For the armour, thermal fatigue and thermal shock resistance, behaviour at transient events such as vertical displacement events, disruptions, and ELMs are the most important factors because significant evaporation, melting, loss of melt layers and cracking may lead to enhanced armour erosion and also to possibility of crack propagation in the heat sink structure.

# Selection of Beryllium grades

- In the ITER Final Design Report 2001, two beryllium materials have been selected:
  - **S-65C Vacuum Hot Pressed (VHP) from Brush Wellman Inc. (USA)**
  - and **DSHG-200 from the Russian Federation.**
- These grades have been selected based on excellent thermal fatigue and thermal shock behaviour, high ductility, low impurity content, an available comprehensive data base (including neutron irradiation effects) and availability.

- **List of considered materials:**

Producer	Grades
Brush Wellman, US	S-65C VHP, S-65C HIP, S-65 CIP, SR-200 VHP, S-200F HIP, S-200F VHP, I-400 VHP
Russian Federation	DShG-200, TShG-56, TR-30, TGP-56, TShGT, DIP-30, TShG-200

(VHP = Vacuum Hot Pressing, HIP = Hot Isostatic Pressing, CIP = Cold Isostatic Pressing)

**ITER R&D program included wide range of studies of mechanical properties, thermal performance at transient events, n-irradiation effects**

# ITER R&D - Selection of Beryllium grades

Various tests simulating disruption, VDE, cyclic loads have been performed with various grades.

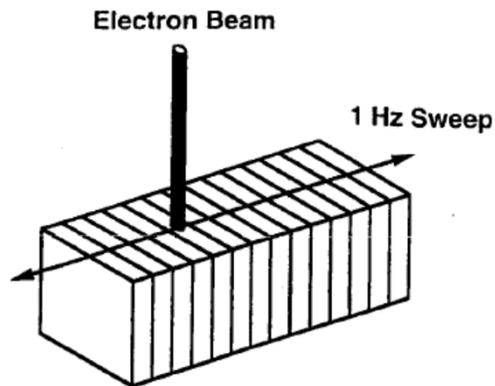
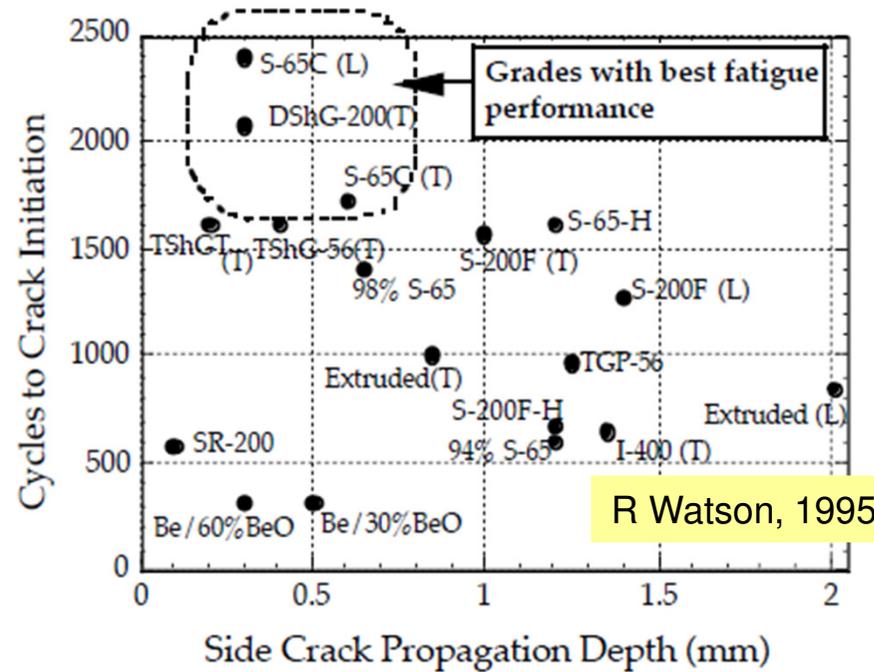


Fig. 1. Conceptual drawing of using a small electron beam spot swept back and forth across the surface of many samples to create low cycle fatigue damage.

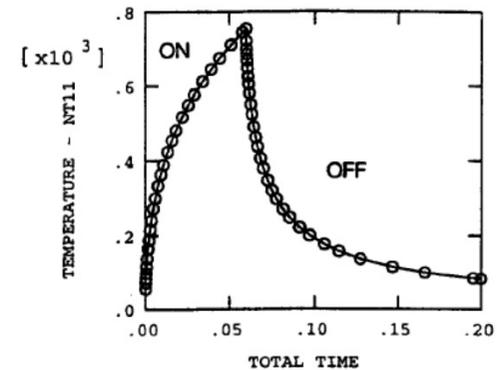
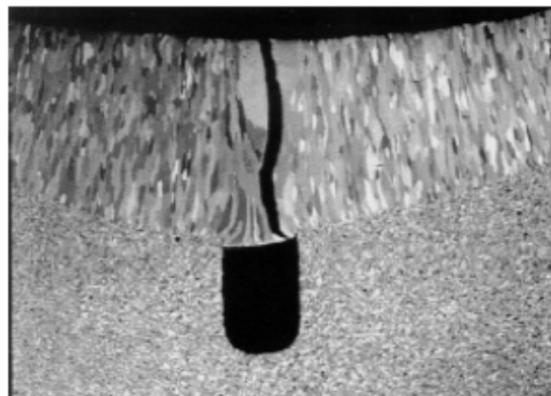


Fig. 5. Temperature (°C) vs. Time (s) for element # 1086. Peak temperature = 750°C at end of heating pulse,  $t = 0.06$  s. Cooldown within 0.2 s to  $< 50^\circ\text{C}$ .

# ITER R&D - Selection of Beryllium grades

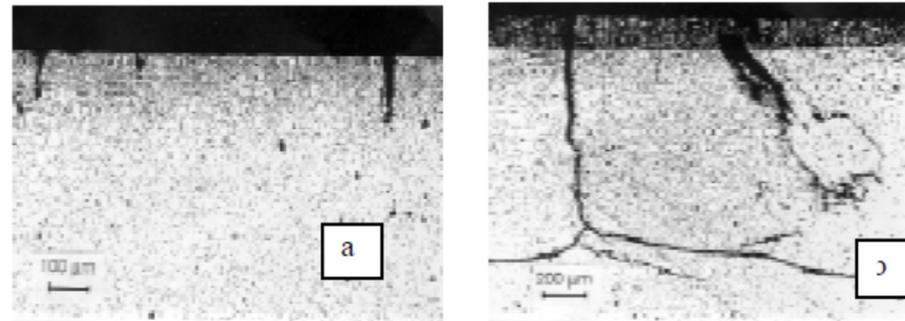
Various tests simulating disruption, VDE, cyclic loads have been performed with various grades.



S65 C / CuCrZr, CuMnSnCe braze  
absorbed energy: 60 MJm<sup>-2</sup>

**Micrograph of a S-65C Armour after  
VDE Simulation at 60 MJ/m<sup>2</sup>**

J Linke, 1997

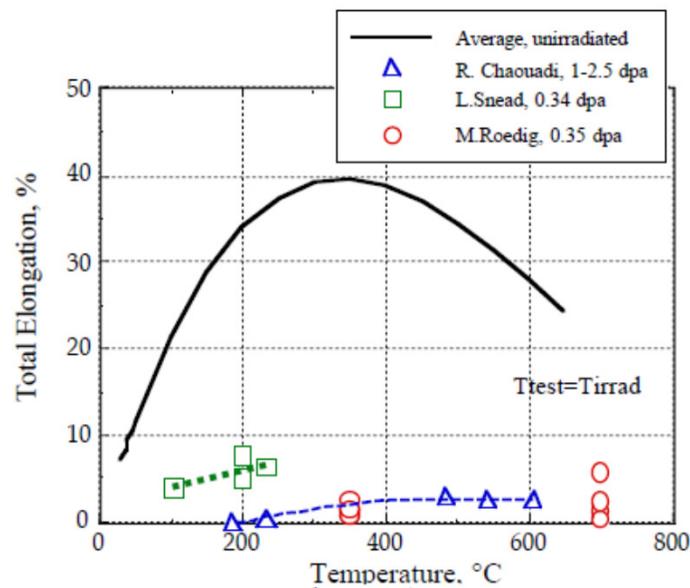


**Metallography of S-65C Be Loaded at Energy Densities of 2.8 MJ/m<sup>2</sup>  
(a) and TR-30 Loaded at Energy Densities of 5.8 MJ/m<sup>2</sup> (b) [16]**

M Roedig, 1995

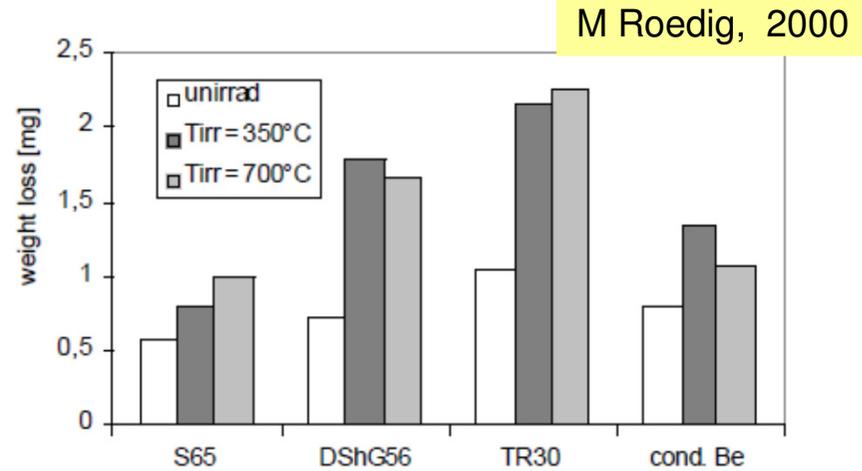
# ITER R&D - Selection of Beryllium grades

Various tests under neutron have been performed with various grades

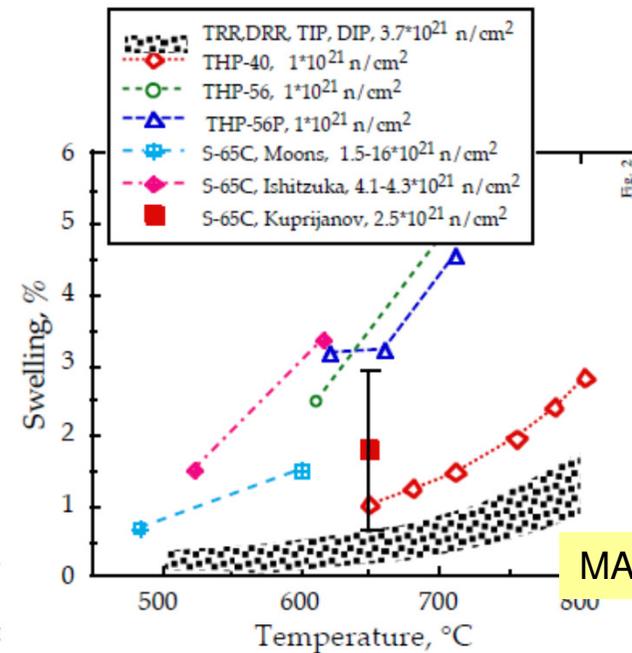


**Total Elongation of the Be S-65C VHP After Neutron Irradiation**

MAR, 2001



**Weight loss During Thermal Shock Tests Disruption Load - 15 MJ/m<sup>2</sup>, 5 ms**



MAR, 2001

# Proposal for new Beryllium grades and Qualification program

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Recently Chinese and Russian ITER Domestic Agencies proposed additional new grades: CN-G01 (from China, CNMC, Ningxia Orient Group Co. Ltd) and TGP-56FW (from Russia, A.A.Bochvar Inst. of Inorganic Materials) for the ITER first wall that will be manufactured in China and Russia.

To assess the performance of these new grades, the ITER Organization, Chinese and Russian Parties established a program to perform the characterization of the proposed materials.

This program included:

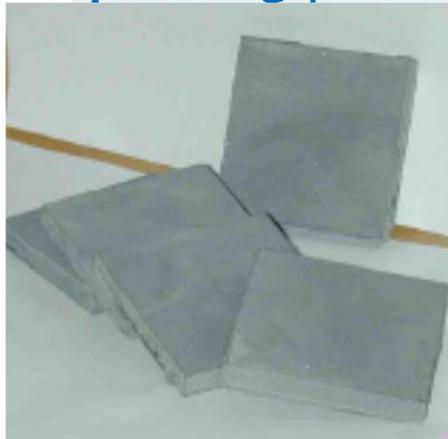
- Characterization of the production technologies,
- Studies of main physical and mechanical properties,
- Comparative thermal performance tests with respect to the grade S-65C

# Qualification of new Beryllium grades

## Manufacturing technologies:

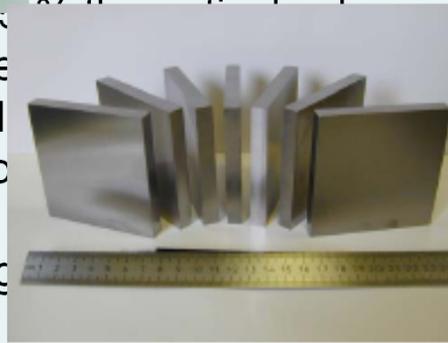
### TGP-56FW

**A modified vacuum hot pressed technique – vacuum hot “packet” pressing (VHPP), Steps:**



Beryllium powder with chemical composition and particle size distribution, after chipping, grinding of ingots, attrition and powder compaction. The density of Be powder is 1.84 g/cm<sup>3</sup>. The density of Be powder in a vacuum hot pressed cross-section up to the density of 7.0-7.3 g/cm<sup>3</sup>.

- Vacuum hot pressed quantity of cold-chamber graphite mold compacted up to density > 7.0 g/cm<sup>3</sup>
- Final machining



### CN-G01

**Powder metallurgy vacuum hot pressed technique. Steps:**

- Beryllium pebbles with purity of 99% are fabricated from beryllium mineral
- Cast into beryllium ingots
- Ingot mechanically chipped into small pieces, which were then impacted into beryllium powders by high speed gas stream.
- Classifying and chemical cleaning,
- VHP beryllium blocks into beryllium powder
- Final machining



# Chemical composition requirements

	Brush Wellman Inc. 1987	Materion Brush Inc. 2011	CN 2010	RF 2010
Product grade	S-65 Revision C	S-65 Revision E	CN-G01	TGP-56 FW
Beryllium Assay, minimum *	99.0	99.2	99.0	99.0
Beryllium oxide	1.0	0.9	1.0	1.0
Aluminium	0.06	0.05	0.06	0.02
Carbon	0.10	0.09	0.1	0.1
Iron	0.08	0.08	0.08	0.16
Magnesium	0.06	0.01	0.06	0.06 (summ for these 4 elements)
Manganese		0.005	0.012	
Copper		0.025	0.012	
Nickel		0.025	0.012	
Chromium		0.01	0.012	0.06
Titanium		0.025		0.04
Zirconium		0.025		
Zinc		0.005	0.008	
Silver		0.005		
Cobalt		0.005		
Lead		0.005		
Calcium		0.005		
Molybdenum		0.005		
Silicon	0.06	0.045	0.06	0.025
<b>Uranium</b>		<b>0.015 (**)</b>	<b>(***)</b>	<b>0.0030</b>
Fluorine				0.005
Other metallic impurities	0.04	0.04 (each)	0.04 (sum of other 15 elements)	
Minimum bulk density (% of theoretical)	99.0	99.0	99.0	99.0
Average grain size, maximum, µm	20	20	20	25

**Weight %, maximum unless specified)**

\* Difference (i.e. 100%-other elements)

## Notes:

**Chemical composition is very similar**

- BeO ≤ 1.0wt.%
- Density min 99.0 wt.%
- Grain size 20 – 25 microns

**ITER Specific safety requirement  
on max U content - 0.0030 wt.%**

\*\* S-65 grade – max 0.015 wt.%,  
but can be easily achieved  
ITER limit

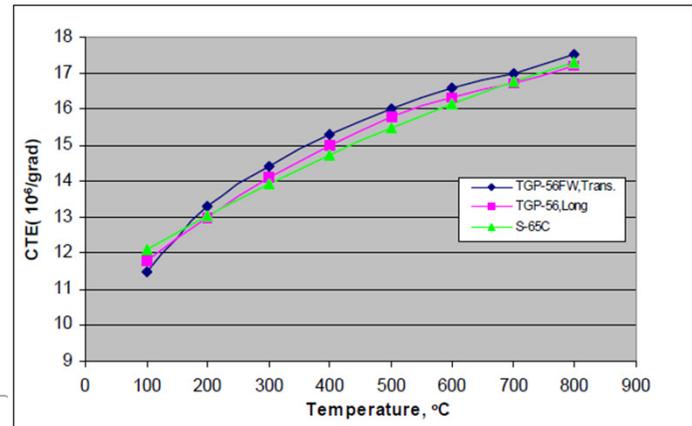
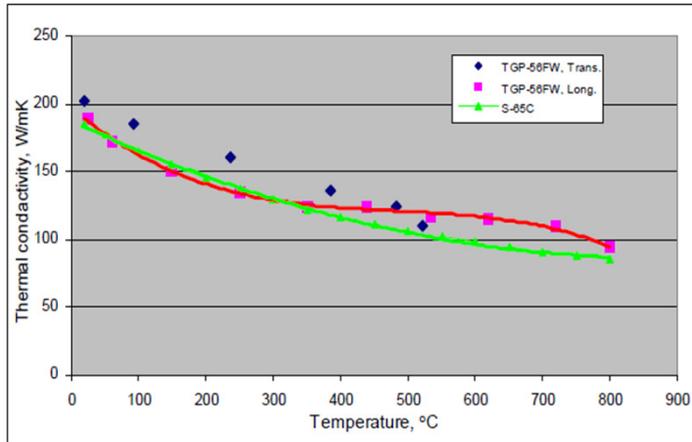
\*\*\* For grade CN-G01 – typical  
measured values < 0.0020 wt.%  
(U is from ore)

# Physical properties characterization

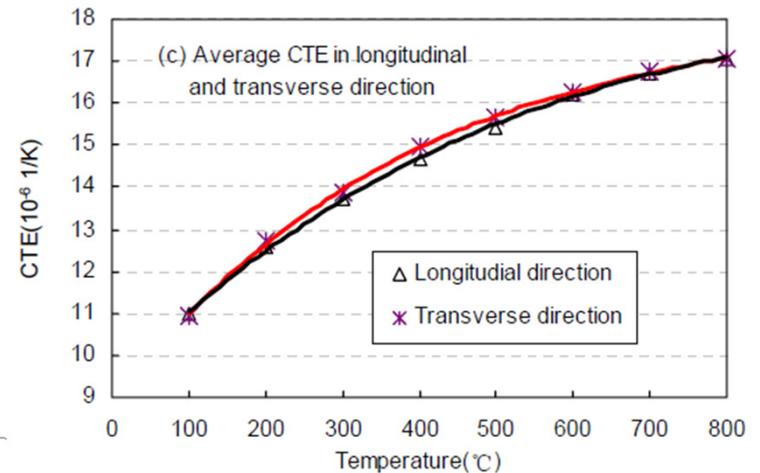
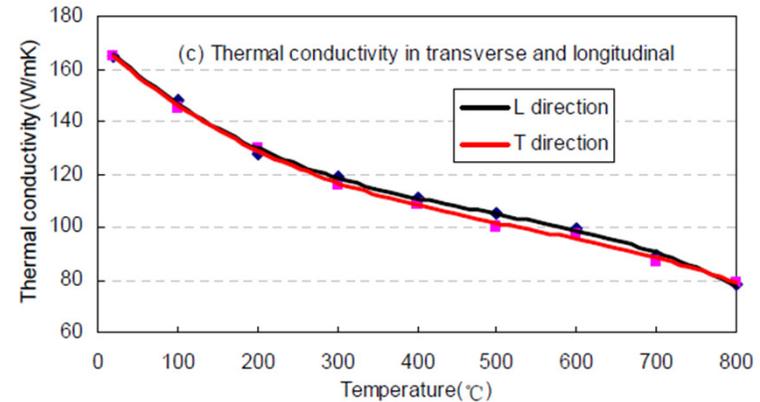
The basic material physical properties [thermal conductivity, coefficient of thermal expansion, elastic modulus, Poisson's ratio, density and specific heat capacity] have been measured

Good agreement with data for S-65C VHP grade has been demonstrated.

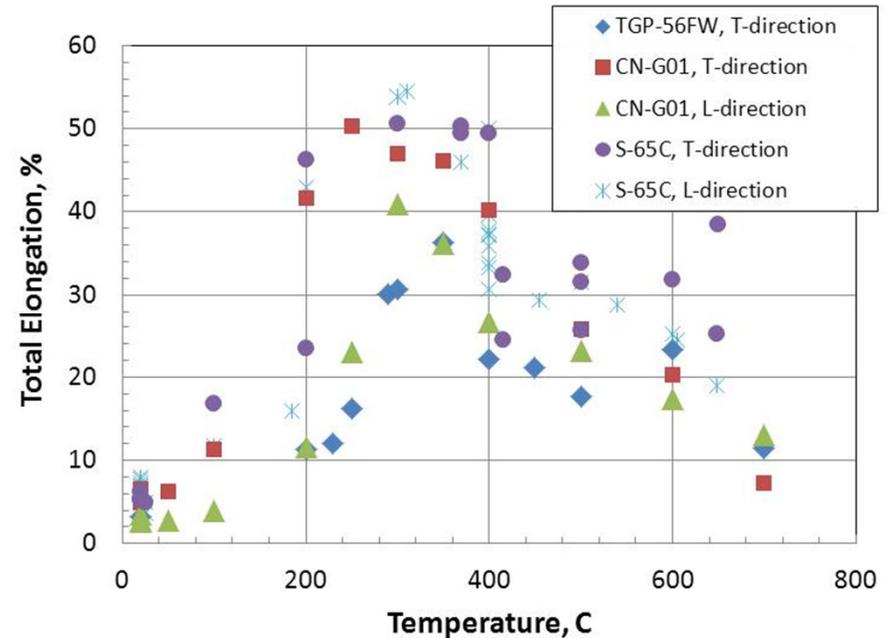
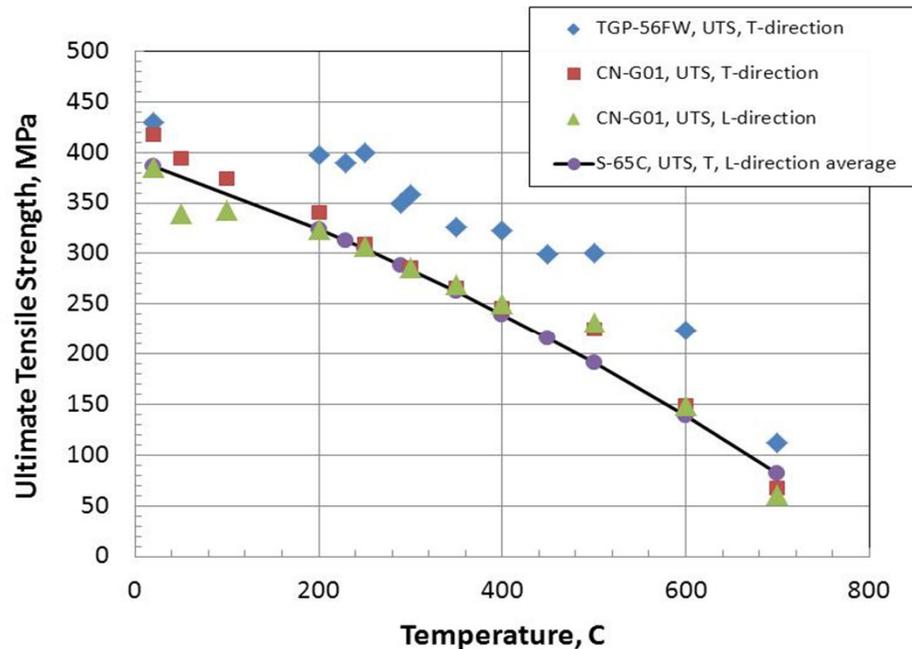
**TGP-56FW**



**CN-G01**



# Mechanical properties characterization



(T- transverse, L – longitudinal to molding pressure directions)

## Ultimate tensile strength and total elongation of S-65C, TGP-56FW and CN-G01 grades

- Min TE for S-65 and CN-G01 is 3% at RT, 2% for TGP-56FW
- Strength is very similar for all grades (slightly higher for TGP-56FW)

# Thermal Performance Qualification

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The program for thermal performance behaviour included several tests such as:

- Thermal shock resistance investigations
- Vertical displacement event (VDE) heat load simulation testing and following thermal shock tests
- Thermal cyclic fatigue tests after VDE simulation testing

The following criteria have been established for acceptance of material after thermal performance tests:

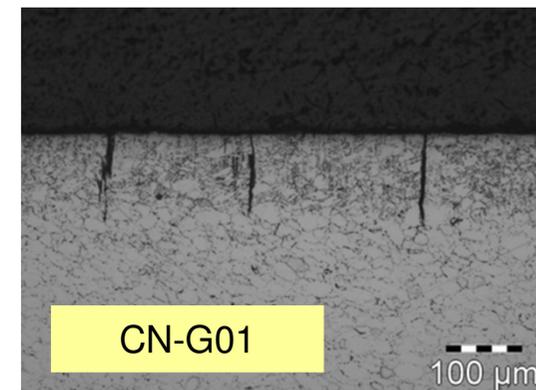
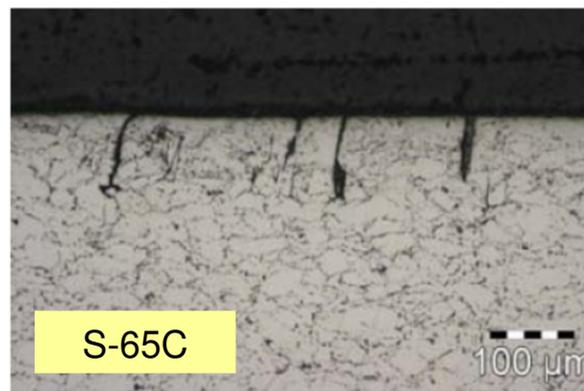
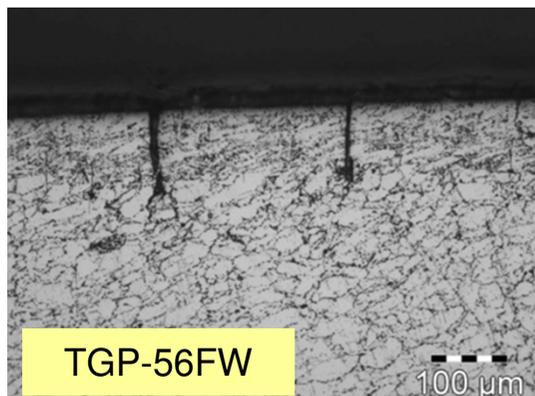
- No cracks in beryllium parallel to the surface (perpendicular to heat direction);
- No macroscopic loss of Be material;

# Thermal Performance Qualification

## *Comparative results of thermal shock resistance*

- A comparative study of TGP-56FW, CN-G01 and S-65C beryllium grades was performed in the **electron beam facility JUDITH-1 (FZJ)**.
- The samples ( $12 \times 12 \times 5 \text{ mm}^3$  and  $12 \times 10 \times 5 \text{ mm}^3$ ) were loaded in a single shot mode between  $1.2$  and  $2.4 \text{ MJ/m}^2$  with a step of  $0.3 \text{ MJ/m}^2$  and also at  $3 \text{ MJ/m}^2$  and  $5 \text{ MJ/m}^2$  loaded are  $5 \times 5 \text{ mm}$ , pulse –  $5 \text{ ms}$ .
- No significant change of weight was observed

### **Metallographic sectioning perpendicular to the loaded surface for the different beryllium grades loaded at $3 \text{ MJ/m}^2$**



M Roedig, 2011

# Thermal Performance Qualification

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## ***Comparative results of multiple thermal shock resistance***

Experiments were performed at room temperature and at 250°C. The loading conditions were the following:

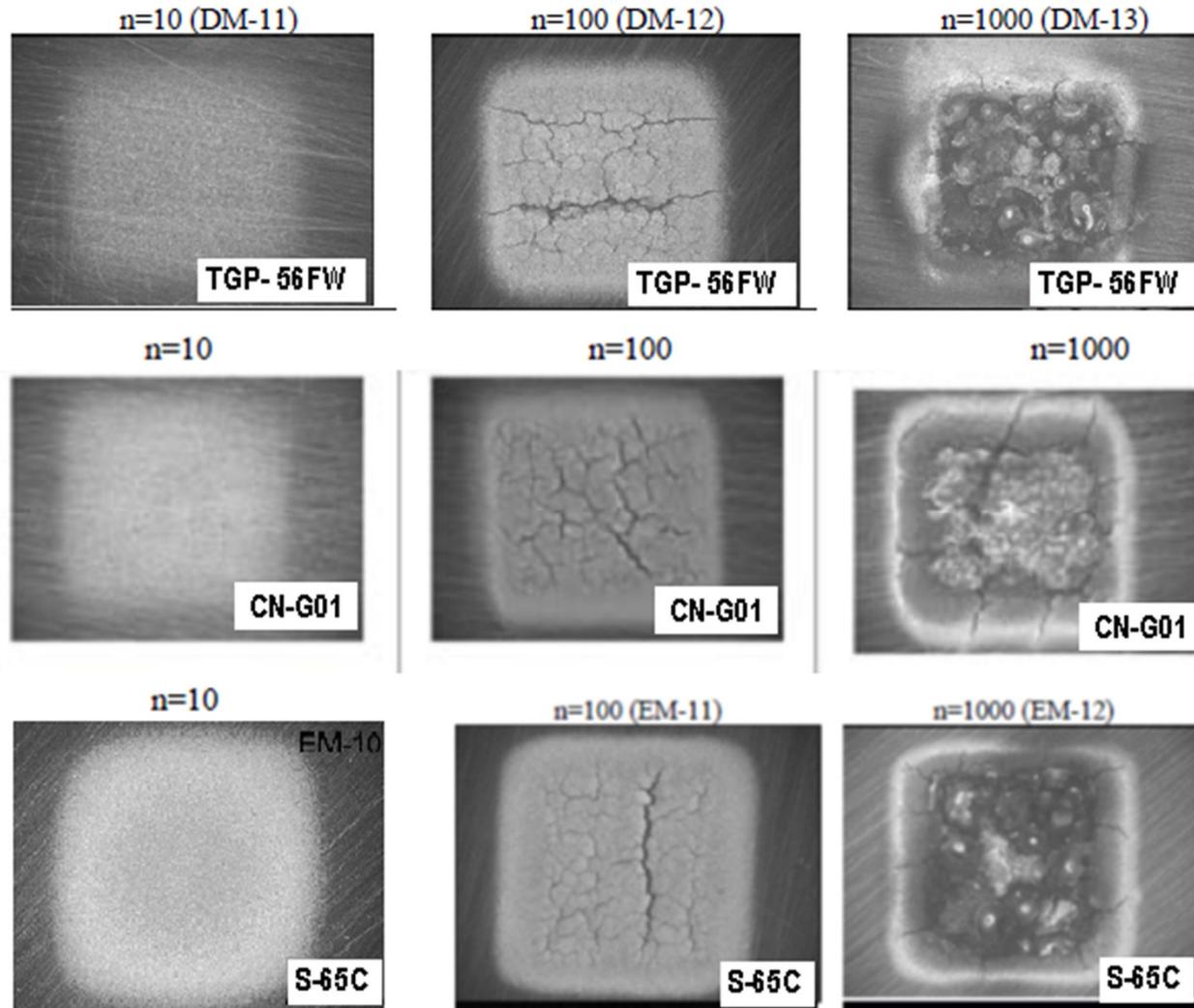
- Energy density - 1.2 MJ/m<sup>2</sup> and 1.5 MJ/m<sup>2</sup>
- Loaded area – 5x5 mm<sup>2</sup>
- Number of shots: 10, 100 and 1000
- Pulse duration - 5 ms.

The main findings can be summarised as following:

- At 1.2 MJ/m<sup>2</sup> and 1.5 MJ/m<sup>2</sup> , no damage after 10 cycles
- After 100 cycles roughening of the surface was observed for all grades
- After 1000 cycles visible crack formation was identified for all grades.

# Thermal Performance Qualification

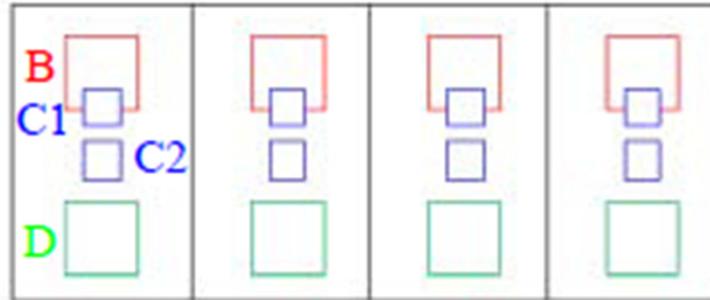
Thermal shot tests with preheated beryllium samples up to 250°C showed a similar tendency but crack formation had been previously observed after 100 cycles and some melting occurs on the surface after 1000 pulses.



# Thermal Performance Qualification

## Comparative results of VDE, disruption simulation and cyclic test

### Loading conditions



**Step A:** screening of the whole surface at  $0.5 \text{ MW/m}^2$

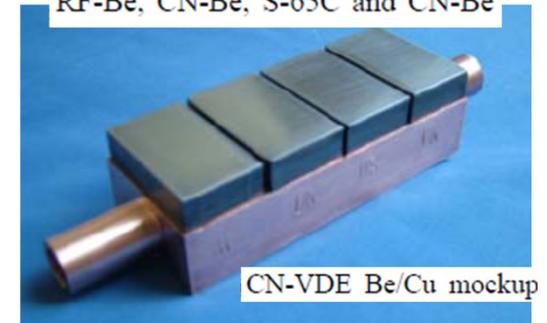
**Step B:** VDE test  
load  $40 \text{ MJ/m}^2$ ,  $a = 10 \times 10 \text{ mm}^2$ , 1 shot  
50 ms ramp-up, 160 ms steady state

**Step C:** disruption test  
load  $3 \text{ MJ/m}^2$ , 1 shot,  $a = 5 \times 5 \text{ mm}^2$ ,  $\Delta t = 5 \text{ ms}$ ,  $P \cdot \nu t = 43$

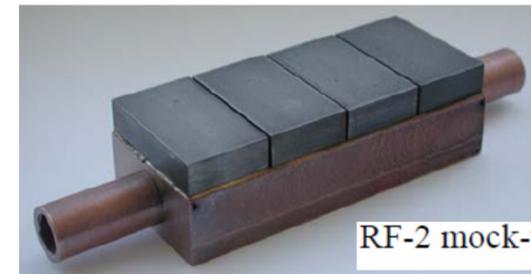
**Step D:** repetitive test  
1000 cycles at  $2 \text{ MJ/m}^2$ ,  $a = 10 \times 10 \text{ mm}^2$ ,  $\Delta t = 25 \text{ ms}$ ,  $P \cdot \nu t = 13$

**Step E:** thermal fatigue (only CN mock-up no. 2 and RF mock-up no. 2)  
1000 cycles at  $2 \text{ MW/m}^2$

RF-Be, CN-Be, S-65C and CN-Be

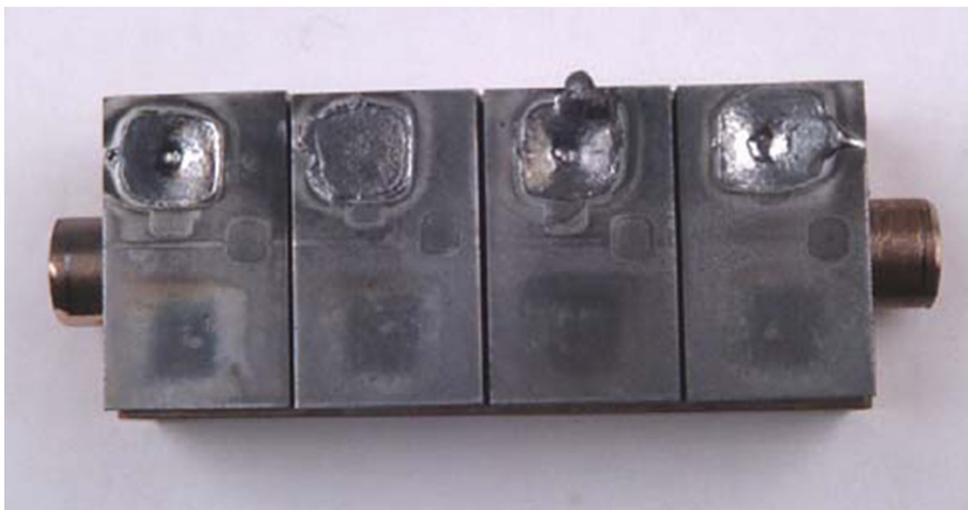


CN-VDE Be/Cu mockup



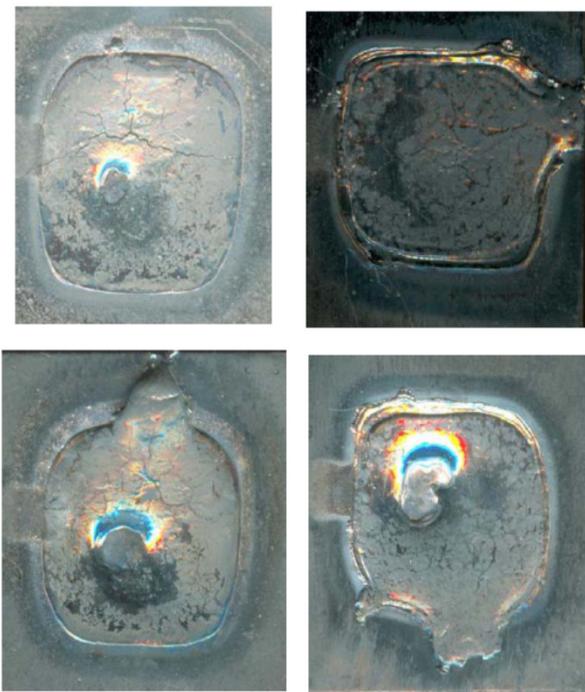
RF-2 mock-up

# Thermal Performance Qualification



A view of water-cooled mock-up after VDE testing, thermal shock testing and thermal fatigue (“Watson-like”) testing (load steps B,C and D)

**VDE test, Load step B,  
40 MJ/m<sup>2</sup>, 1 shot, 10x10 mm**

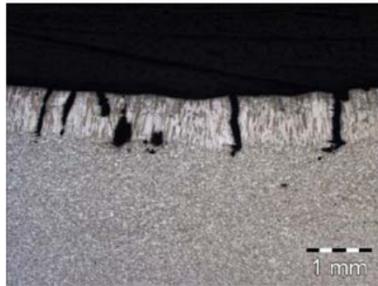


TGP-56FW (1.0 % BeO, trans)

S-65C (long)

See poster P73B, Kupriyanov et al for details

# Thermal Performance Qualification



a

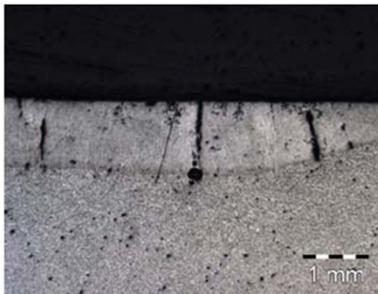


b

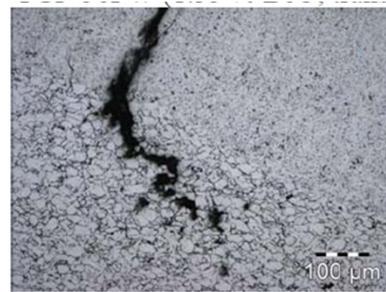


c

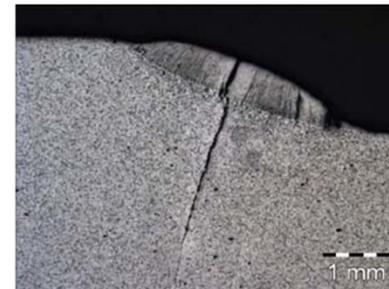
TGP-56FW (1.0 % BeO, trans)



g



h



i

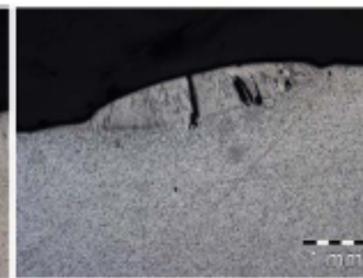
S-65C (long)  
Modul FT163-2

Modul FT163-2

CN beryllium



20304  
Ziegel 2 (B)  
Werkstoff: Beryllium



20258  
Ziegel 4 (B)  
Werkstoff: Beryllium

In most cases the cracks ended at the melting layer only in few cases cracks propagated deeper and this was observed for all grade

See poster P73B, Kupriyanov et al for details

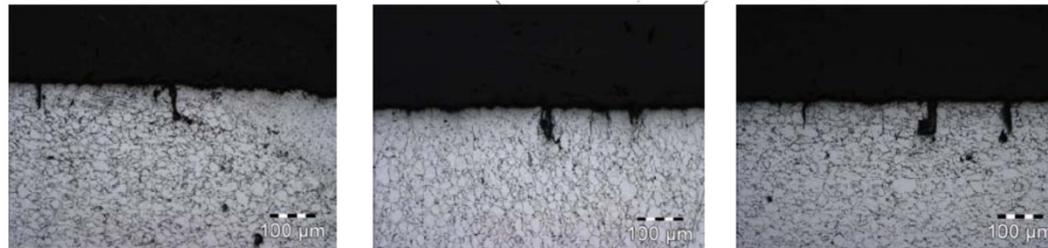
# Thermal Performance Qualification

*Thermal shock testing (load steps C1, C2: 3 MJ/m<sup>2</sup>, 1 shot, a = 5 × 5 mm<sup>2</sup>, 5 ms)*

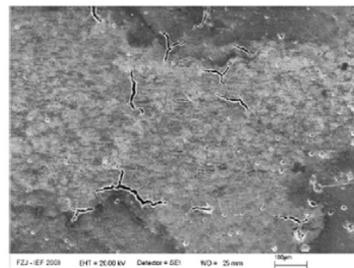
The melting and cracking behaviors are very similar for the TGP-56 FW, CN beryllium and S-65C samples



TGP-56FW (1.0 % BeO, trans)



S-65C (long.)



FT163/1  
tile 2 - spot C1  
RD75B02



CN beryllium

# Thermal Performance Qualification

***Thermal fatigue (“Watson-like”) testing (load step D: 1000 shots at 80 MW/m<sup>2</sup> (2 MJ/m<sup>2</sup>), a = 10 × 10 mm<sup>2</sup>, t = 25 ms)***

- The cracking behaviour of grades is very similar. In few tile large cracks were observed, due to faulty shot and surface preparation.
- After repeating of test on specially prepared set of tiles, behavior of grades was the same.

***Thermal cyclic fatigue test (load step E: 1000 cycles at 2 MW/m<sup>2</sup>, a = 24 × 40 m, t cycle = 15 s heating/15 s cooling)***

- The effect of thermal cycling fatigue loading (1000 cycles at 2 MW/m<sup>2</sup>) on the growth of cracks, which formed on previous stage of testing, was not detected for all tested materials.

**The general conclusion is that behavior of proposed grades is very similar at selected testing conditions**

# Summary

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- Additionally to selected reference grade S-65 VHP (production of Materion Brush Beryllium & Composites, USA) two new grades have been proposed for the ITER First Wall application:
  - TGP-56FW - Russian Federation (Bochvar Inst. of Inorganic Materials)
  - CN-G01 - China (CNMC, Ningxia Orient Group Co. Ltd)
- The detailed characterizations of these grades, comparative behavior of all grades at various types of heat loading conditions have been performed.
- It was concluded that the proposed Chinese (CN-G01) and Russian (TGP-56FW) beryllium grades can be accepted
- Three grades of beryllium are now available for the application of armour for the ITER first wall, S-65, CN-G01 and TGP-56FW.