UT-VIV/PFC-Damage

TASK-TITLE: STUDY OF DAMAGE MECHANISMS IN PLASMA FACING COMPONENTS

INTRODUCTION

Plasma facing components (PFC) for future fusion reactors have to withstand high heat fluxes. In case of TORE SUPRA, the developed components were made of a high thermal conductivity CFC material (a composite made with carbon matrix reinforced by carbon fibres) mechanically and thermally bonded to a copper heat sink and able to remove incident stationary heat flux of 10 MW/m^2 [1]. In order to reach a value of 20 MW/m^2 for the divertor component of the ITER machine, the lifetime of this assembly submitted to considerable thermal stresses must be increased. The objectives of this activity are:
(i) to provide a study of damage mechanisms of the CFC bond,
(ii) to propose an optimization of the bond and
(iii) to develop a model for predicting the lifetime of the bond under operating conditions.

2005 ACTIVITIES

During this period, the actions foreseen were achieved:

Realisation of mechanical tests on CFC samples in order to identify their constitutive law [2]

Various mechanical tests are necessary in order to identify the damageable constitutive law of the CFC. During this period, a shear test procedure has been developed and validated in order to characterize the non linear shear behaviour of the CFC (figure 1).

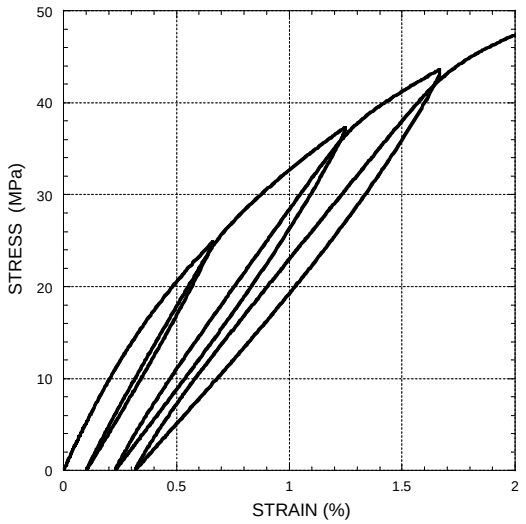
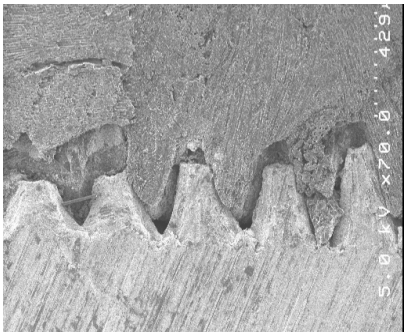


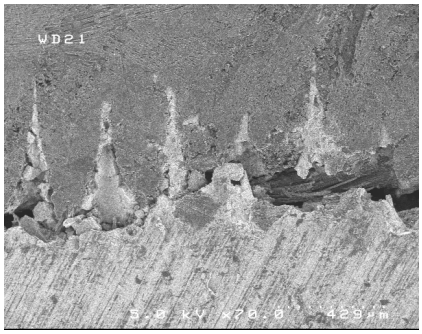
Figure 1: Mechanical response of a HL1 carbon/carbon composite in shear

Observations of damaged tiles to locate damage mechanisms [3]

Damaged tiles obtained from components tested under high heat flux were submitted to micrographic observations. Two damage modes of the interfacial zone were clearly evidenced (figure 2): debonding of the interface and failure of the copper spikes.



a)



b)

Figure 2: The two different mechanisms of failure of the CFC/Cu interface of a PFC tested under high heat flux:
a) debonding of the copper spikes,
b) failure of the copper spikes

Interfacial crack propagation modelling [4], [5]

Numerical simulations were performed to analyse the initiation and the propagation of the interface failure (figure 3). An initiation criterion was used in order to predict the initiation of a crack at the edge of the component as a function of the interfacial properties (figure 4a). A damage model (cohesive zone model) of the interface was identified by comparing modelling results with experimental values obtained from tensile (figure 4b) and shear tests of the bond.

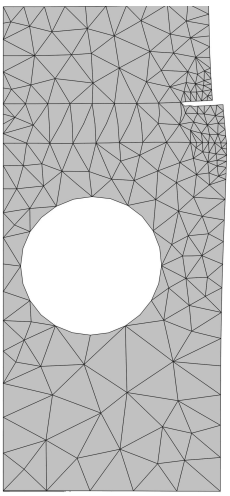


Figure 3: Modelling of the interface : Initiation and propagation of a crack at the CFC/Cu interface

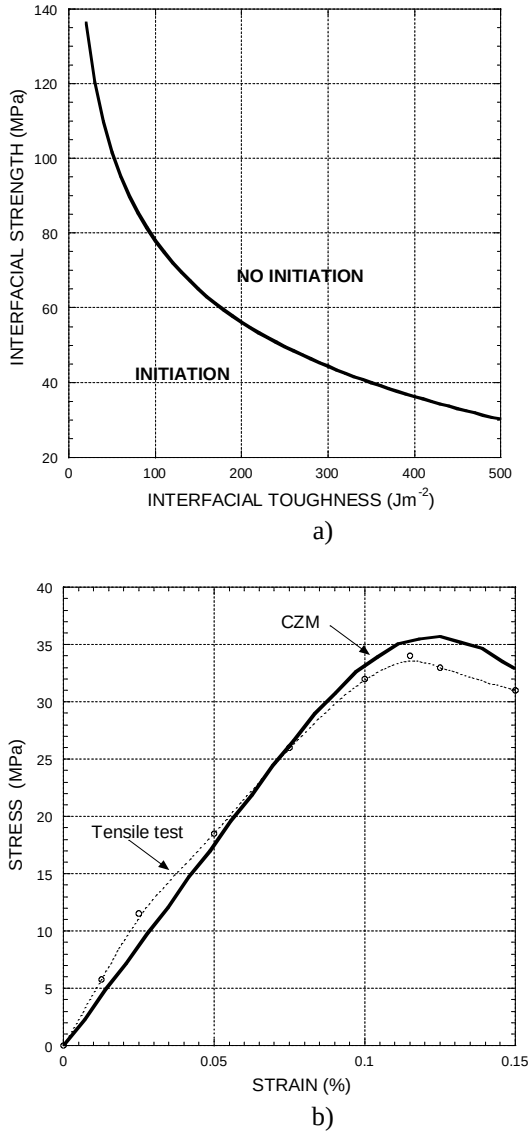


Figure 4: Modelling of the interface
a) Initiation domains as a function of the interfacial strength and toughness
b) Identification of a damage model of the interface with the help of a tensile test on a CFC/Cu specimen

CONCLUSIONS

As detailled before, in 2005, the actions concerning:

- Realisation of mechanical tests on CFC Samples in order to identify their constitutive low;
- Observation of damages tiles to locate damage mechanisms;
- Interfacial crack propagation modelling has been successfully achieved.

The work will continue in 2006 with
(i) tests for thermal expansion measurements of the constituants of the component,
(ii) mechanical tests on CFC samples,
(iii) prediction of the life time of the bond under operating conditions.

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UT-VIV/PFC-HIP

Task Title: IMPROVEMENT OF RELIABILITY, PERFORMANCE AND INDUSTRIAL RELEVANCY OF HIP PROCESSES FOR PFC COMPONENTS

INTRODUCTION

The fabrication of many fusion reactors components is based on the Hot Isostatic Pressing (HIP) technique. The use of HIP as a means for diffusion welding and complex components fabrication is not so frequent, and there is a need to improve the industrial relevancy of these processes. Four subjects have been identified: machining and cleaning of surfaces for diffusion welding, modeling for powder compaction, and, for both processes, outgassing and tool and anti-diffusion materials. Prospective routes have been studied in 2004 which needed further experiments to finalize all parameters influence.

2005 ACTIVITIES

PROPERTIES OF JOINTS

Experimental work has been performed with 316L or 316LN forged material and 316LN powder.

Influence of machining and HIP temperature

Solid/solid joints

In 2004, the influence of roughness on the impact toughness of solid joints was studied. The HIP temperature was 1040°C. Materials were cleaned following the reference laboratory route. The joint impact toughness (KCU) was 99, 96 and 83 J/cm² for joints made with blocks milled to Ra in the range of 0.8, 3 and 6 µm respectively. Surprisingly, the joint impact toughness was only 45 J/cm² for joints made with ground blocks (Ra~0.3 µm). In order to assess the effect of temperature similar samples were HIPed at 1100°C. A drastic improvement is noticed: the joint impact toughness increased up to 221 J/cm² for fine milled surfaces and 122 J/cm² for ground surfaces. Joint examination reveals that sets of inclusions are evenly spread along the joint in the "ground sample", while no such feature can be observed in the "milled sample" (figure 1).

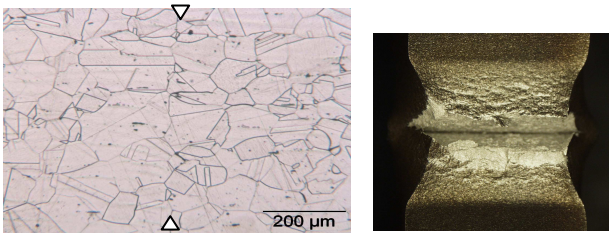

Figure 1: Microstructure of SS316L joints HIPed at 1100°C and associated KCU rupture surface fine milled surfaces.

It is concluded from these results that the grinding process deteriorates the surfaces. It is known that grinding is a very energetic process that can led to overheating of the surfaces when materials with poor thermal properties are considered.

Powder/solid joints

Experiments achieved with solid/solid joints show that the 1100°C HIP cycle associated with the reference laboratory surface preparation and a fine milled surface condition gives the best results. However, in many instances the combination of machined blocks with powder has large advantages in terms of fabrication, such like for the ITER shield modules. Because the manufacturing costs of such a large component largely depend on machining, the question arises whether requirements as stringent as the 'fine milled condition' are necessary or not for solid/powder joints. Solid/powder joints were elaborated with forged blocks milled to various roughness values. Blocks were cleaned following the reference laboratory surface preparation route, cans were filled with powder and HIPed at 1100°C. Tensile tests were achieved on the joint elaborated with a block Ra=0.15-0.20 µm. Rupture occurred on the solid part of the specimens (figure 2). RCC-M requirements were fulfilled.

Figure 2: Solid/powder joint tensile samples after testing. Joint location is indicated by small lines.

Impact toughness testing gave the following values: 191; 198, 200 and 196 J/cm² for surfaces machined to Ra=0.15-0.2, 0.8, 1.6 and 3.2 µm respectively (figure 3). It is concluded that the surface roughness is not very important for solid/powder joints. It seems that these joints do not require specific machining requirements.

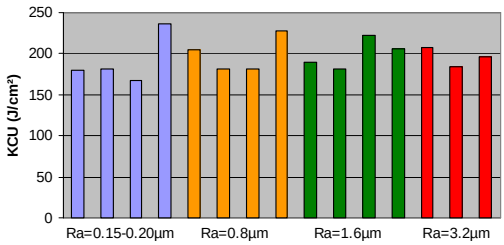

Figure 3: Impact toughness of solid/powder joints HIPed at 1100°C depending on the surface roughness of the solid part.

Influence of cleaning process

At the present time, a manual operation is carried out to clean the surface of the pieces at a laboratory scale. More industry-relevant solvent cleaning methods have been considered because they can be applied for a large variety of materials and component shapes. Perchloroethylene and hydrofluoroether tested in 2004 gave lower performance than our laboratory manual method, but the cleaning conditions were perfectible.

A collaboration has been made in 2005 with an industrial partner proposing cleaning methods using particular solvent products in industrial equipment. A test program has been identified on the base of similar applications.

Anti-diffusion materials

Anti diffusion materials are used when the joining of two parts needs to be avoided. The two main questions to be assessed are the efficiency of the anti-diffusion materials and their influence on the joint performance. In 2004, various foils and felts have been tested as anti-diffusion materials. The cans comprised a joint and a tool. The tools were easily removed but the joint impact toughness was very low, less than 50J/cm².

In 2005, experiments were made with oxide coatings (thick plasma sprayed alumina, thin PVD coatings of SiO_2, Al_2O_3 and TiO_2) which were thought to be less polluting. All cans were HIPed at 1040°C to enable comparison with preceding results. In all cases, the removal of the tool was possible, though more difficult than with foils and felts, but the joint impact toughness were again low. Then, nitride-base PVD coatings have been tested because of their stability in the tested temperature range.

In this case, the tool could not be removed. A metallographic examination revealed a non-uniformity of the coating thickness with local lack of nitride layer where a welding between the disc and the tool occurred (figure 4).

Table 1: Effect of anti-diffusion materials on joint impact toughness.

Description	*Thickness*	*KCU (J/cm²)*
Plasma sprayed alumina	350 µm	60±6
Physical Vapour Deposited Silicon oxide	>3 µm	30±3.5
Physical Vapour Deposited Alumina	>3 µm	14±8
Physical Vapour Deposited Titanium oxide	>3 µm	29±5
Physical Vapour Deposited TiN	~ 3 µm	-
Physical Vapour Deposited TiN/TiAlN nanolayers	~ 3 µm	-

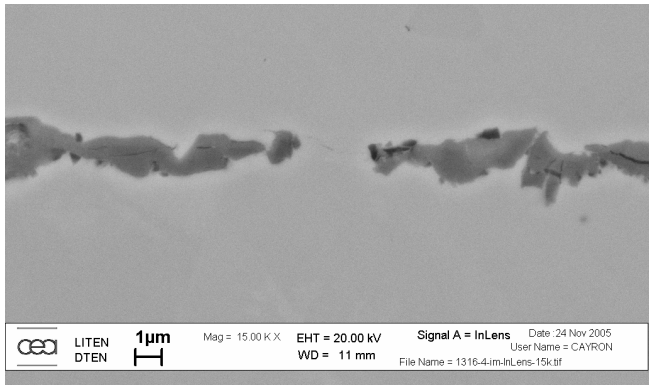

Figure 4: Failure of anti diffusion coating in 316L/316L joint, leading to undesired local diffusion bonding

POWDER OUTGASSING

Powder metallurgy is an attractive way to manufacture complex components at low cost with a good precision. However, consolidation of powder by HIP process depends strongly on the oxide layer at the powder particle surface. The presence of oxygen compounds inside the powder such as adsorbed water molecules can lead to the growth of the oxide layers during the HIP processing at high temperature and thus drastically reduce the mechanical strength of parts manufactured with HIPed powder. In our study we have investigated several experimental conditions to treat 316LN powder before HIP cycles. Two experimental routes have been studied. The first one enables to measure the influence of N_2 flushing gas to treat 316LN powder confined inside a low conductance vessel where high vacuum is difficult to achieve. This configuration leads to small vacuum treatment and poor mechanical results. 316LN specimens manufactured with N_2 flushing treated powder presents low impact KCU toughness (between 100 J/cm² and 120 J/cm² for powder HIPed at 1100°C, 140 MPa). The second experimental set-up enables the heat treatment of the powder under high vacuum level (under 10-5 mbar). At this pressure, the high temperature treatment of the powder (from 140°C to 200°C) gives good mechanical results. Impact KCU toughness higher than 160 J/cm² is obtained for all the samples manufactured with 316LN powder heat treated above 140°C. Best results (toughness of about 180J/cm²) are obtained for temperatures close to 200°C.

MODELING

Concerning simulation, we have adjusted a novel law for powder densification during HIP (ref. 316_POU9) based on in-situ dilatometry experiments. The novel law is an Abouaf law with addition of some isotropic hardening in the 316LN constitutive law. The identification has been done on in-situ dilatometry experiment results (figure 5) and on the deformation of a simple bi-material mock-up (figure 6) that exhibits some bending after HIP (figure 7). Physical thermo-mechanical coupling through the relative density and temperature has also been implemented: the thermal parameters depend upon the relative density according to a law adapted from the law proposed by Argento [1].

A parameterized study has also permitted to appreciate which parameter of the law is important with regard to the phenomenon to be modelled. This will help in designing and identifying the next generation of constitutive laws.

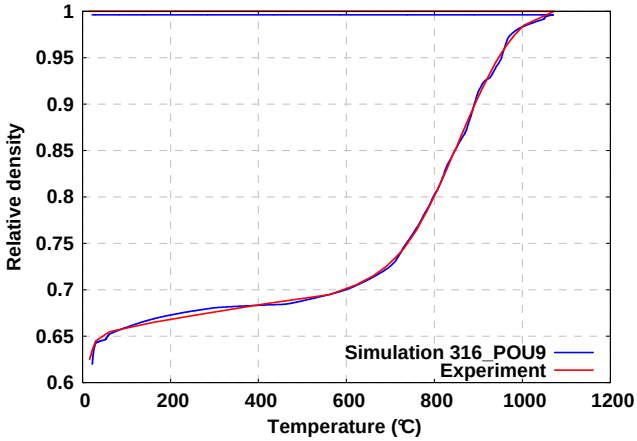

Figure 5: Adjustment of the powder law on in-situ dilatometry densification curve

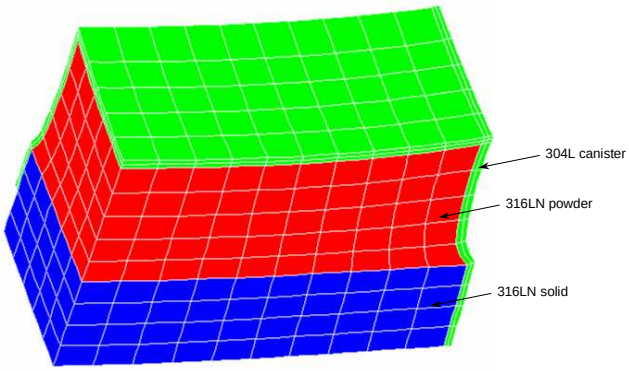

Figure 6: Mesh and deformed shape of the modelled quarter of the SEMU0.
Dimensions:200 x100 x (50 powder + 30 316LN) mm

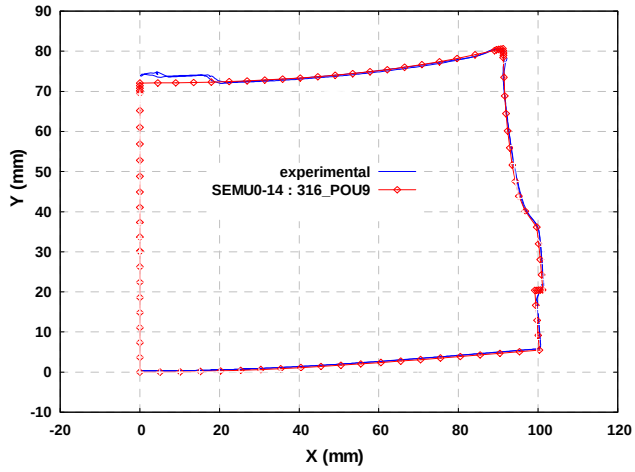

Figure 7: Shape of the SEMU0 on its symmetrical plane along its longer dimension. Comparison to simulation obtained with 316_POU9 law is also shown

CONCLUSIONS

Specifications are now available for the elaboration of 316LN solid and solid to powder joints. Unfortunately, no efficient industrial surface preparation route has been found up to now. Experiments with anti diffusion materials are disappointing too. As far as powder HIP is concerned, the link between the outgassing procedure, the amount of oxygen in the powder material and its impact toughness has been established. The powder outgassing procedure is now improved. Finally, significant improvement in finite element modelling of powder HIP compaction has been made by adjusting calculation laws.

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UT-VIV/PFC-NanoSic

Task Title: NANOCRYSTALLINE SILICON CARBIDE (SIC) MECHANICAL PROPERTIES OF NANOCRYSTALLINE SIC

INTRODUCTION

The task concerning nanocrystalline silicon carbide (SiC) is divided in two parts related respectively to the optimization of the preparation of dense nanocrystalline silicon carbide (SiC) by Hot Isostatic Pressing (HIP) and to the evaluation of the mechanical and thermal properties. The first part of the task has been completed in 2004 with the getting of densification rates of 95 % of the theoretical density (TD) for samples sintered without sintering additives and of 98 % for those sintered with additives. Grain sizes obtained for samples sintered without sintering additives were around 50 nm or below whereas for samples sintered with additives, grain sizes between 50 and 100 nm were observed. In 2005, mechanical properties were measured on the samples sintered during the first half of the task. The properties were measured on samples obtained after HIP or after HIP + Post-HIP thermal treatments. The Post-HIP treatments were performed with the aim to follow the evolution of some mechanical properties with the grain size in the materials. Hardness and fracture toughness were measured at room temperature and flexural strength and Young's modulus were measured both at room and at high temperatures.

2005 ACTIVITIES

MECHANICAL PROPERTIES AT ROOM TEMPERATURE: HARDNESS, YOUNG'S MODULUS AND FRACTURE TOUGHNESS

Vickers hardness

The hardness has been measured by the indentation method. This method consists of indenting the tested materials with a diamond indenter, in the form of a right pyramid with a square base and angle of 136° between opposite faces subjected to a load of 1 to 100 kgf. The Vickers Hardness is calculated by measuring the two diagonals of the indentation left in the surface of a material after removal of the diamond indenter. The table 1 presents the results obtained for samples immediately after the sintering by HIP.

The hardnesses of the samples sintered by HIP are in the range 15-18 GPa. These values are smaller than those reported in the literature [1], [2] for micro / sub micrometric SiC (20 to 25 GPa) but are in agreement with an inverse Hall-Petch effect (decrease in strength with a decrease of the grain size) which could come from relaxation processes taking place at the grain boundaries and resulting in a decrease of the strength below a critical grain size [3]. This inverse Hall-Petch effect has been reported in literature for SiC with an optimum grain size for hardness of 100-200 nm [4]. In order to follow the evolution of the hardness with

grain size, post-HIP thermal treatments were performed at 1650°C, 1750°C, 1800°C and 1850°C under argon during 1 hour. The figure 1 shows the effect of the temperature of the post-HIP treatment on the hardness. A strong increase (up to 28-29 GPa) is observed after a post-treatment at 1750 °C for XXIII 20 and XXIII 22. These values are higher than the best hardness known for polycrystalline microstructured SiC (25 GPa) and also higher than the best hardness reported in literature for nanocrystalline SiC sintered by HIP (27 GPa) [4]. It is expected that the post-treatments promote grain growth in the range 100-200 nm favorable to the getting of optimum values for hardness.

Table 1: Vickers Hardness (HV 2) for samples after sintering by HIP (1930°C – 190 MPa -1h30)

Sample	Sintering aids (wt. %)	Density (% TD)	Grain size (nm)	Vickers Hardness (GPa) - HV2
XXIII 19	0	93	11	16
XXIII 20	8 (YAG)	96	90	17
XXIII 21	8	97	62	17
XXIII 22	5	97	-	18
XXIII 23	2	98	19	15
XXIII 24	0	95	-	16

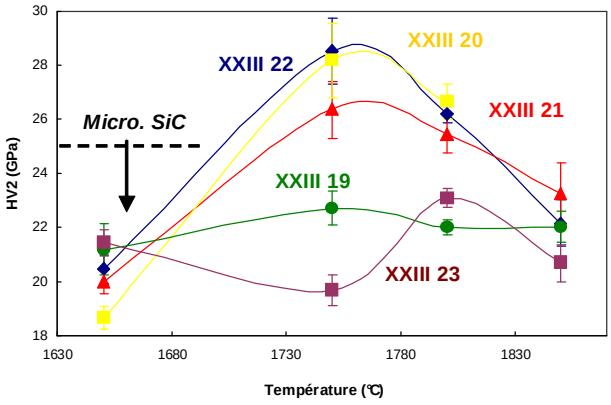

Figure 1: Evolution of the hardness as a function of the Post-HIP temperature

In order to correlate post-HIP temperatures to grain sizes, a Hall –Williamson (HW) analysis was performed on the X-Ray diffraction patterns recorded on post-HIPed samples. The results are given in table 2 for the samples containing 8 wt. % of sintering additives (XXIII 20 and XXIII 21).

The results confirm that after a post-HIP thermal treatment at 1750°C, the grain sizes of the samples are in the range 100 - 200 nm where maximum values of hardness are observed (see figure 1). Furthermore, the results of grain size determination reveal that it is possible to change at will the mean single crystal size in the sintered samples in the nanometric / sub-micrometric domains and consequently the mechanical properties.

Table 2: Single crystal sizes estimated in post-HIPed samples from X-Ray diffraction patterns

Reference	Post-HIP temperature (°C)	Density (% of TD)	Single crystal size (nm)
XXIII 20 (8 wt. %) YAG	1650°C – 1h – Ar 1750°C – 1h – Ar 1800°C – 1h – Ar	92 95 95	76 177 331
XXIII 21 (8 wt. %) Eutectic	1650°C – 1h – Ar 1750°C – 1h – Ar 1800°C – 1h – Ar 1850°C – 1h - Ar	93 96 96 95	79 136 270 283

Young's modulus

The measurements of Young's modulus were performed by the "impulse excitation technique" on a GrindoSonic equipment. The results of Young's modulus are presented in table 3.

Measurements were performed on HIPed samples and on few ones post-HIPed. Globally, the values are located between 235 and 322 GPa and they are smaller than those reported in literature for microstructured SiC sintered with 2 wt. % of alumina and yttria additives by hot pressing (414 – 445 GPa) [1].

An explanation could be the high proportion of the grain boundaries phases (amorphous glassy phases with quite lower Young's modulus) in our samples coming from the high proportion of silica [5] (silica has a Young's modulus of only 73 GPa).

Fracture toughness

The facture toughness has been estimated from hardness and Young's modulus measurements by measuring size of the cracks generated at the extremities of the diagonal of the Vickers imprint. The fracture toughness of the samples is reported in table 3. The values of fracture toughness are lower than those of classical SiC (around 4.5 MPa $m^{1/2}$) [1] excepted for the sample XXIII 20 (composition of the YAG phase at grain boundaries) for which fracture toughness is 5.9 MPa $m^{1/2}$ after a post treatment at 1750°C. This result is in agreement with previous work [6] reporting high fracture toughness for samples sintered with a mixture of sintering aids (Al_2O_3 and Y_2O_3) in proportions favorable to the formation of the YAG phase at grain boundaries after sintering.

Table 3: Young's modulus (E in GPa) and fracture toughness (K1C in MPa $m^{1/2}$) for HIPed and few post-HIPed samples.

Sample	HIP		HIP + 1650°C		HIP + 1750°C		HIP + 1850°C	
	E	K1C	E	K1C	E	K1C	E	K1C
XXIII 19	235	1.9	-	-	-	-	-	-
XXIII 20	322	4.2	-	-	260	5.9	-	-
XXIII 21	303	2.5	-	-	-	-	-	-
XXIII 22	300	2.2	300	2.1	245	2.2	300	2.4
XXIII 23	252	3.7	-	-	-	-	-	-

MECHANICAL PROPERTIES AT HIGH TEMPERATURE: YOUNG'S MODULUS AND FLEXURAL STRENGTH

Young's modulus up to 1000°C

The measurements of Young's modulus at high temperature were performed in a furnace under an argon atmosphere on a parallelepiped attached with a thread to alumina supports (figure 2). Excitation is performed with an alumina missile propelled by an argon jet.

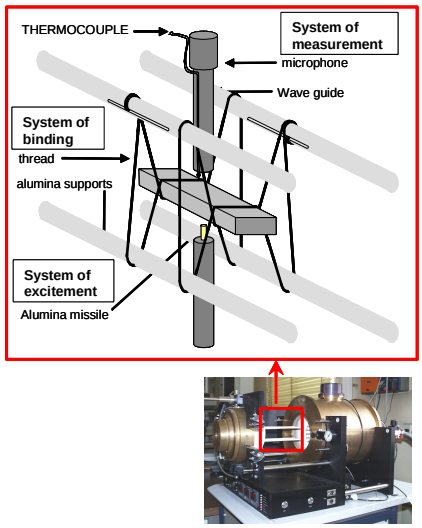

Figure 2: Device for the measurement of Young's modulus at high temperature by the impulse excitation technique

Results of Young's modulus measurements with temperature are presented on figure 3.

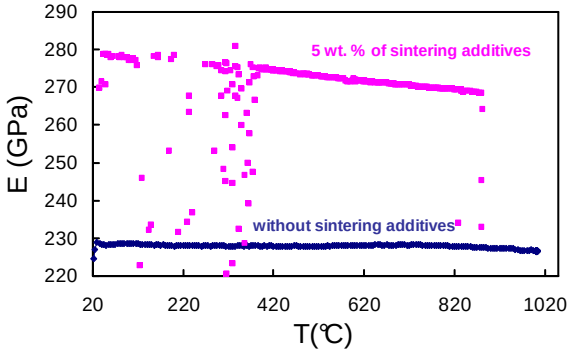

Figure 3: Evolution of the Young's modulus with temperature for two samples.

The measurements were performed on one sample sintered without sintering additives and on one sample sintered with 5 wt. % of sintering additives (Al_2O_3 and Y_2O_3) at the eutectic composition. At first, the comparison of the two samples reveals that the Young's modulus values are higher for sample containing sintering additives than for sample sintered without additives. For the sample sintered without sintering additives, a very low decrease is observed up to 1000°C (- 1 %) whereas for the sample containing 5 % of sintering additives a more important continuous decrease is observed (- 4 %) at 800°C. The more important decrease observed for the sample sintered with additive could come from sintering additives which influences the behaviour at high temperature. Indeed, Young's modulus measurements performed on pure Al_2O_3 and pure Y_2O_3 revealed a continuous decrease of the modulus of approximately 10 % at 1000°C [7], this behaviour being consistent with the decrease observed for the sample sintered with additives.

Bending Strength up to 1200°C

Four-point method has been applied to determine flexural strength at room and high temperature on 3 samples. The bend tests were performed at CEA – Grenoble. The results are plotted on figure 4 and compared to the results found in literature [8] for sub-micrometric SiC (> 540 nm) sintered by hot pressing with Al_2O_3 and Y_2O_3.

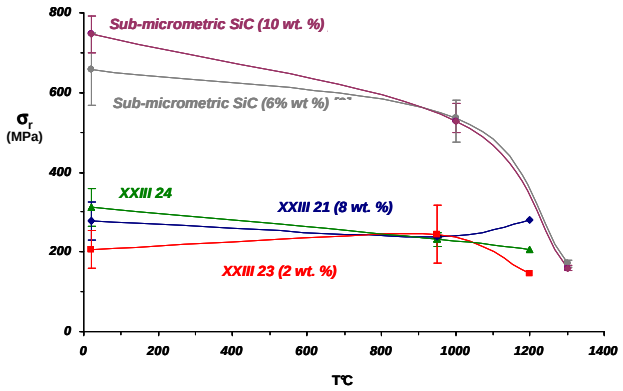

Figure 4: Evolution of the four-point bending strength of the samples with temperature. Results concerning sub-micrometric SiC are also given [7]

The bending strength values obtained for nanostructured SiC are smaller than those observed for sub-micrometric SiC [8] but the stability seems to be better especially after 800°C. The decrease with temperature observed beyond 1000°C for sub-micrometric SiC has been interpreted by Sciti et al. by the softening of the phase at grain boundaries. On the basis of this interpretation, a similar behaviour should be observed in our samples but the behaviour seems quite different. Until now, this result is not well understood and additional experiments are needed to confirm these preliminaries observations.

CONCLUSIONS

In conclusion, the activity performed in 2005 revealed the possibility to adjust at will the grain size in HIPed SiC nanoceramics in the nanometric/sub-micrometric ranges by performing post-HIP thermal treatments between 1650 and 1850°C. By doing so, an optimum value of 28-29 GPa for the hardness has been reached for grain sizes of 100 - 200 nm as expected from literature data. To our knowledge, these values are the highest never reported in literature for a monolithic SiC elaborated by powder metallurgy. The maximum value of hardness corresponds also to the maximum value for fracture toughness (5.9 MPa $m^{1/2}$). At high temperature, a good stability of the Young's modulus and of the bending strength seems to occur, especially for the samples sintered without sintering additives but additional experiments are needed to confirm the preliminaries observations.

In 2006, the investigation of the mechanical and thermal properties at high temperature will be continued (Young's modulus, thermal conductivity). Creep tests will be performed and the effect of cyclic and/or long thermal treatments on the nanostructure will be investigated. The Thermal shock resistance could be measured on some samples.

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UT-VIV/PFC-Pyro

Task Title: APPLICATION OF A TRICOLOUR PYROREFLECTOMETER TO PLASMA FACING COMPONENTS IN-SITU INFRARED MONITORING

INTRODUCTION

The main subjects presented here are the following:

1) Comparison of an optical fibre probe equipped with a reflecting hemisphere which had been used for the measurements in 2004 with a new flat-headed one.
   Determination of the emissivities (that could not be measured in 2004) versus temperature by a direct method for the wavelength 1.3 µm and 1.55 µm.
2) Presentation of the development state of the dedicated solar set up DISCO and the tricolor pyroreflectometer for testing fusion material including a dedicated probe.
3) Preliminary tests for remote measurements on FE 200 and Tokamak

2005 ACTIVITIES

DEFINITION OF A PROBE ADAPTED TO DIRECT EMISSIVITY MEASUREMENTS

The experimental set up used is MEDIASE [2] as presented in the report A2 [1] and the sample tested (diameter. 25 mm, thick. 2 mm) is a pure W sample from Plansee Gmbh delivered by CEA [1].

These complementary tests are implemented for two reasons:

1) to compare results obtained with a probe equipped with a reflecting hemispherical probe -previous tests A2 - and a dedicated flat probe which avoids the interferences due to the multiple reflections (figure (1)).

2) to determine the emissivities at 1.3 and 1.55 µm through a direct method according to (1).

$$\varepsilon(T,\lambda,\theta) = L^{\circ}(Tr(\lambda,\theta))/\ L^{\circ}(T,\lambda). \qquad (1)$$

Tr, is the measured radiance temperatures.

T is the true temperature of the sample that is assumed to be equal to the convergence temperature T* determined with a pyroreflectometry method [3] based on the introduction of a diffusivity factor η

$$\eta(T) = \rho^{0,\cap}(\lambda,T) / \rho^{0,0}(\lambda,T) \qquad (2)$$

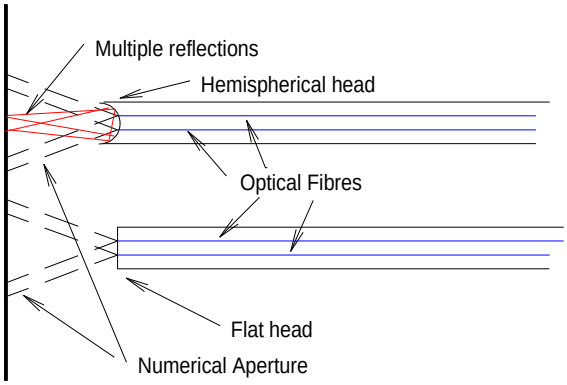


Figure 1: Scheme of the two kinds of probes equipped with optical fibres.

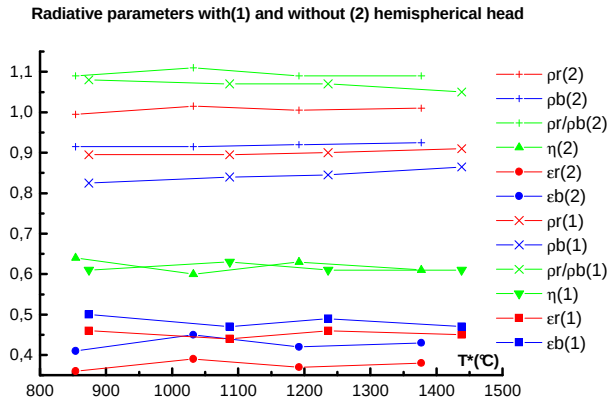


Figure 2: Results obtained with hemispherical (1) and with flat-headed(2) probe on a W sample
ρ normal normal reflectivity, ε normal emissivity,
η diffusivity factor
r corresponds to measurement at 1.55 µm, b to 1.3 µm.

The figure 2 shows the thermoradiative parameters measured with a two color pyroreflectometer: symbol (1) is used for the hemispherical probe measurements, (2) for the flat-headed probe measurements.
With the hemispherical head the emissivity is more important and the reflectivity is less important than with the flat head. This is the logical effect of the multiple reflections between the surface sample and the reflecting coating of the probe.
The ratio between reflectivities is almost constant. So the use of a flat or hemispherical probe is indifferent in this respect of the ratio.
Despite the fact that the diffusivity factors are equal in this case, only the flat probe gives good values for reflectivity and emissivity.

Table 1: Flat probe results on W during a thermal cycle

T* °C	Trr °C	Trb °C	ρr	ρb	εr	εb	η	ρr/ ρb
871	737	767	1.475	1.36	0.34	0.39	0.45	1.08
868	738	768	1.48	1.355	0.35	0.41	0.44	1.09
1050	883	921	1.48	1.355	0.37	0.42	0.43	1.09
1189	992	1036	1.495	1.37	0.37	0.43	0.42	1.09
1370	1120	1171	1.49	1.385	0.37	0.41	0.43	1.08
1625	1298	1357	1.565	1.48	0.36	0.4	0.41	1.06
1016	842	878	1.61	1.505	0.33	0.37	0.42	1.07

The table 1 summarises the results obtained with the flat probe configuration on the same sample as used for the measurements shown in figure 2 during one thermal cycle inside the solar furnace of the MEDIASE device.

Since W has specular reflection properties the normal reflectivities are larger than the value one.
The normal emissivities are in accordance with literature results [4].

DEDICATED APPARATUS DEVELOPMENT STATE

Two specific pieces of equipment have been developed in the frame of this task a tricolor pyroreflectometer and a dedicated vacuum vessel, the solar device DISCO.

The tricolor pyroreflectometer:

Components needed for the pyroreflectometer have been collected with difficulties and only now the realization of the apparatus is in the last phase with a large delay. Fortunately, the two color technique is enough to validate the method.

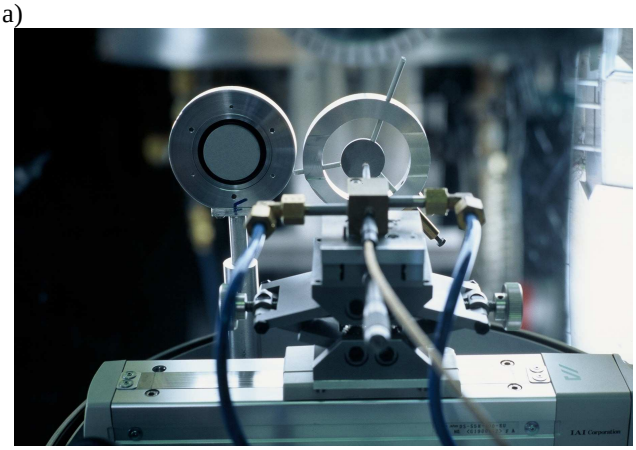
The dedicated solar device:DISCO (DIspositif Solaire de Caractérisation Optique i.e. Solar facility for Optical Characterization)

The specific vessel DISCO is finished and the photos of the figure 3 illustrate the set up.

The left one presents a detail with a water cooled probe – originally designed for the FE 200 test device, before it became obvious that another approach is needed (see section IV) - in front of a W sample back-face and next to it a reflectance reference for comparison measurements.

The photo (a) shows the vessel at the focus of a solar installation with a front window and a front-face of the sample.

a)



b)

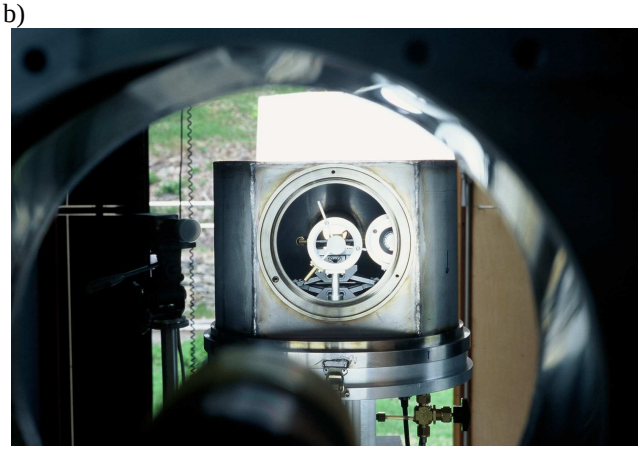


Figure 3: Photos of the dedicated vacuum vessel DISCO

Until now DISCO has been used for two kind of characterizations:

- to validate the measurement of the convergence temperature T* from an angular position different to the normal one.

- to measure the Bidirectionnal Reflection Distribution Function (BRDF) with a normal incident illumination.

i) Measurements under non-normal angles (preparation for FE 200)

The idea is to realize pyroreflectometric measurements on FE 200 in a configuration outlined in figure 4. In this case the location and orientation of the probe is angular out of the destroying electron's beam zone.

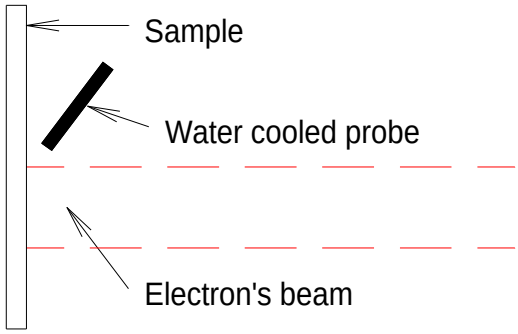


Figure 4: Scheme of a possible measurement configuration on FE 200

The table 2 summarises the results obtained when the axis of the probe is placed at different angles to the normal position of the sample.
The only measured parameters are the reflectivity, the others are simulated.
We can observe that the measured reflectivity decreases normally with the angle. However the ratios between the two reflectivity measurements rest the same within the accuracy of the measurement (better than 2%). By simulation we can estimate the convergence temperature T* and we conclude in accord with a theory [3]: the method is applicable because the ratio ρr/ ρb is constant.
30° is the limiting angle for this kind of measurement - for larger angles the measured signal is too low.

ii) BRDF determination for W samples

BRDF determination has been realized with a multi optical fibre probe. The observing fibres are located at 0, 10, 20, 30, 40, 50 and 60°. (figure 5)

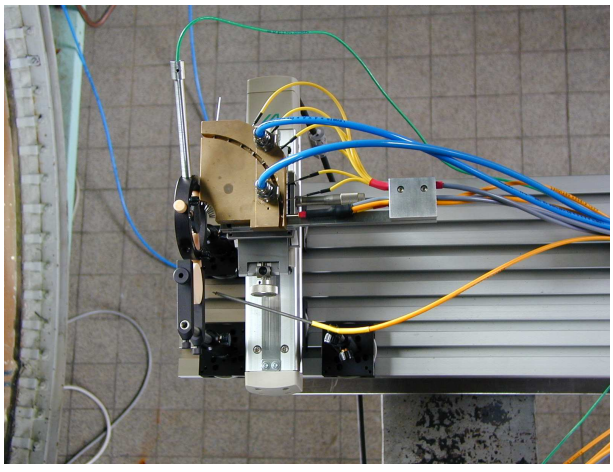


Figure 5: Photo of a BRDF measurement probe installed on DISCO

The results obtained show (figure.6) the specular properties of the W samples.

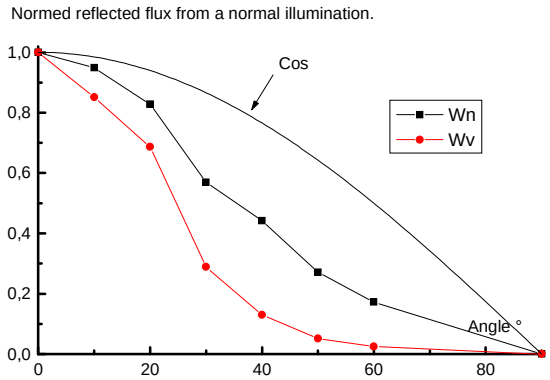


Figure.6: BRDF for W clean (Wn) and W glass-blasted (Wv)

PRELIMINARY WORK FOR REMOTE MEASUREMENTS.

The risks generated by the use of a water cooled probe on F200 near the electron's beam zone and the configuration for measurements in tokamaks directed the studies in the direction of the development of components and apparatus able to remote measurements from a distance of 1 m from the target.
To this purpose we have evaluated the measurement range of:

- a commercial monochromatic pyroreflectometer

- a commercial laser diode actually used in IMP pyroreflectometer systems.

i) Test of a commercial pyroreflectometer: Quantum Logic Model QL3600C-1A

Quantum Logic Model QL 3600C-1A is a device dedicated to measurements on diffuse samples. For samples with lambertian behavior, it delivers the temperature and the emissivity of the observed surface at a distance of 0.6 m and at the wavelength 0.9 µm.

The use of this kind of apparatus can be useful for the control of plasma facing components.
We have realized several measurements on different surfaces to evaluate the performance of the Quantum device for emissivity and indirectly reflectivity determination.

The table 3 shows all the results obtained.

The Quantum pyrometer appears adapted to measurements on diffuse surfaces. The use of its technology for the ITER configuration and the convergence method need improvement in optics and signal acquisition and treatment. It needs particularly the adjunction of a second wavelength.

Table 2: R esults for different angular measurements on W

Angle°	ρr	ρb	ρr/ ρb	εr	εb	η	T* °C
0	1.51	1.41	1.07	0.32	0.37	0.45	1000
15	1.15	1.08	1.06	0.31	0.35	0.60	1006
30	0.22	0.20	1.1	0.36	0.42	2.91	980

Table 3: Results obtained with Quantum pyroreflectometer
(Diffuse samples are lambertian reflection standards with reflectivity values as indicated)

Sample	Diffuse 98	Diffuse 75	Diffuse 50	Diffuse 20	Diffuse 10	Cu blasted	Cu polished	W clean	W oxidized
ε(0.9 µm)	0.5	0.27	0.52	0.80	0.89	0.20	0.33	Out of scale	0.50

ii) Distance with a commercial laser diode

A laser diode (characteristics: λ =0.830µm and power =1 W) has been tested without focalisation optic in the actual IMP configuration probe.
The reflected signal obtained at 0.60 m is about 80 mv and about 40 mV at 1 m.

The laser diodes of the present version of pyroreflectometer used at Odeillo (λ 1.3 µm and 1.55 µm) have a power of less than 100 mW. Consequently the improvement of the tricolor pyroreflectomer to extend its distance of observation needs the development of a dedicated optic and some preliminary measurement tests.

CONCLUSIONS

Pyroreflectometry method, probe and apparatus were tested on PROMES facilities to be improved for measurements on CEA reactors and facilities as FE200 or Tokamaks:

- A probe with a 'flat head' has been compared to an 'hemispherical head'. Only the flat probe has allowed to determine correctly the emissivities at 1.3 µm and 1.55 µm for W sample delivered by CEA.

- The device DISCO has been used to validate pyroreflectrometric measurements with an orientation of the probe in a direction different than the normal (to the surface) position.

- This configuration can be applicable to FE 200 but unfortunately the risks of damage from the electron's beam impedes real tests. Complementary measurements have been realized to determine the B.R.D.F. of W samples.

- Preliminary tests have been conducted for remote measurements. Two technical solutions appear:

- An improvement of a commercial solution.

- A design and a realization of an optical head adapted to the IMP fibre optical pyroreflectometer.

The second solution offers the advantage to continue with the same architecture and to benefit of the laboratory developed software and methods.

If the power of the presently used laser diode should not be enough, it may become necessary to change the used wavelength. All these technologic improvements and tests are the challenge for 2006.

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