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ITER ECRF Window Development
- CVD-Diamond Window -

- Final Report -
ITER Task No.: G 55 TT 19 EU (TWO-ECRF/WIN)

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Executive Summary:

In accordance with the goals of this ITER Task experimental and theoretical investigations on 1 MW, 170 GHz, CVD-diamond gyrotron and torus windows for continuous wave (CW) operation have been continued.

Two gyrotron window units were tested in collaboration with the Russian Hometeam. Unfortunately, both window disks cracked after operation at mm-wave power levels around 1 MW and pulse lengths up to 1.5 s. The reasons are only known for the second unit where a sag of the window disk (120-140 μm), introduced by its brazing, led to a static stress of approximately 0.7-0.8 MPa. A third CVD-diamond disk unit does not show any sag. The high power tests are scheduled for January 2002.

A CVD-diamond window disk was irradiated with a neutron fluence of almost 10^{21} n/m^2 and then brazed to two Inconel waveguide cuffs. The window unit has been mounted into a test housing in an evacuated corrugated HE_{11} waveguide (I.D. = 63.5 mm) and successfully tested in collaboration with the Japanese Hometeam at mm-wave power levels of 0.58 MW (0.2 MW) and pulse length 15 s (132 s). The temperature rises of the disk at a diameter of 65 mm and at the disk water cooling rim were 27°C and 2°C, respectively. Simulation calculations show agreement with the measured temperature rise curves for $\tan\delta = 4.8 \cdot 10^{-5}$ and k = 1200 W/mK which reveals that the reduction of the thermal conductivity by a factor of 2 after neutron irradiation was partially removed during the brazing at temperatures above 600°C. A 118 GHz CVD-diamond torus window unit was successfully in collaboration with CES Cadarache at a mm-wave power level of 0.3 MW and pulse durations up to 111 s.

Dielectric, thermal and mechanical properties of a series of CVD-diamond window disks manufactured by DeBeers (Charters, UK) and FhG-IAF (Freiburg, D) have been analyzed. The permittivity at room temperature always is $\varepsilon_r = 5.67 \pm 0.01$ and the loss tangent at the disk centers $\tan\delta = 1\text{-}\ 2 \cdot 10^{-5}$. Only a small variation with temperature has been observed up to 700 K. Surface losses introduced by the brazing process are still being investigated. Irradiation by a neutron fluence of almost 10^{21} n/m^2 increaded the loss tangent to $4 \cdot 10^{-5}$ but did not change ε_r. Thermal conductivity was found to be strongly reduced (840 W/m · K as compared to 1800-2000 W/m · K for unirradiated CVD diamond). The mechanical strength (growth face under tension) is 400 (± 30) MPa and has not been weakened by the neutron irradiation mentioned above.

ITER ECRF Fensterentwicklung
- CVD-Diamantfenster -

ITER Task No.: G 55 TT 19 EU (TWO-ECRF/WIN)
- Schlussbericht -

Kurzfassung:

In Übereinstimmung mit den Zielen des ITER Programmpunktes wurden sowohl experimentelle als auch theoretische Untersuchungen zu 1 MW, 170 GHz, CVD-Diamant-Gyrotron- und Torusfenster, welche im Dauerbetrieb (CW) betrieben werden können, fortgesetzt.

Zwei Gyrotron-Fenstereinheiten wurden in Zusammenarbeit mit dem Russischen Hometeam getestet. Unglücklicherweise brachen beide Fensterscheiben, nachdem sie bei Millimeterwellenleistungen von ungefähr 1 MW und Pulslängen bis zu 1.5 s betrieben wurden. Die Gründe sind nur für die zweite Einheit bekannt, bei der eine von dem Einlötvorgang herrührende Durchbiegung der Fensterscheibe (120-140 μm) zu einer statischen mechanischen Spannung von 0.7-0.8 MPa führte. Eine dritte Fenstereinheit zeigt keinerlei Durchbiegung der Diamantscheibe. Sie soll im Januar 2002 Hochleistungstests unterzogen werden.

Eine CVD-Diamant-Fensterscheibe wurde mit einer Neutronenfluenz von nahezu 10^{21} n/m^2 bestrahlt und danach an zwei Inconel-Hohlleiterstücke gelötet. Die Fenstereinheit wurde in eine Testfassung in einem evakuierten, korrigierten HE_{11}-Hohlleiter (I.D. = 63.5 mm) montiert und in Zusammenarbeit mit dem Japanischen Hometeam bei Millimeterwellenleistungen von 0.58 MW (0.2 MW) und Pulslängen von 15 s (132 s) erfolgreich getestet. Der Temperaturanstieg der Scheibe bei einem Durchmesser von 65 mm bzw. am wassergekühlten Rand der Scheibe betrug 27°C bzw. 2°C. Simulationsrechnungen stimmen mit dem beobachteten Verlauf des Temperaturanstiegs für $\tan\delta = 4.8 \cdot 10^{-5}$ und k = 1200 W/mK überein. Das heißt aber, dass die durch die Neutronenbestrahlung herrührende Verkleinerung der Wärmeleitfähigkeit um einen Faktor 2 durch die Erwärmung beim Löten bis über 600°C teilweise ausgeheilt wurde. Eine 118 GHz, CVD-Diamant-Torusfenster-Einheit wurde in Zusammenarbeit mit CEA Cadarache bei einer Millimeterwellenleistung von 0.3 MW und Pulsdauern von bis zu 111 s ebenfalls erfolgreich getestet.

Dielektrische, thermische und mechanische Eigenschaften einer Serie von der Fa. DeBeers (Charter, UK) und vom FhG-IAF (Freiburg, D) hergestellter CVD-Diamant-Fensterscheiben wurden analysiert. Die Permittivität bei Raumtemperatur ist immer $\varepsilon_r = 5.67 \pm 0.01$ und der Verlusttangens im Scheibenzentrum ist $\tan\delta = 1\text{-}2 \cdot 10^{-5}$. Dabei wurde nur eine geringe Erhöhung der Werte bis zu Temperaturen von 700 K beobachtet. Durch den Lötprozess bedingte Oberflächenverluste werden weiter untersucht. Bestrahlung mit einer Neutronenfluenz von nahezu 10^{21} n/m^2 erhöhte den Verlusttangens auf $4 \cdot 10^{-5}$, veränderte aber ε_r nicht. Die Wärmeleitfähigkeit jedoch wurde dabei stark verringert (840 W/m · K statt 1800-2000 W/m · K für unbestrahlten CVD-Diamant). Die mechanische Festigkeit (Wachstumsseite unter mechanischer Spannung) ist 400 (± 30) MPa und wurde durch die Neutronenbestrahlung nicht geschwächt.

Contents

1 Report on the Test of a 1 MW, 170 GHz, CW, CVD-Diamond Gyrotron Window

1.1 Method of mounting MPACVD-diamond windows

Metallization/bonding techniques for MPACVD-diamond disks have been developed in a collaboration of FZK Karlsruhe with DeBeers. The elastic properties of a ductile Al-based braze joined by solid-phase diffusion bonding to the diamond disk are used to reduce the thermal stressing as much as possible. The disadvantage of this technique is the relatively low allowed maximum bakeout temperature of the window unit of 450°C (guaranteed by DeBeers) and the danger of corrosion. Several test windows were thermally cycled as if they would be installed as a window on a gyrotron. No diffusion degradation of the brazing has been observed. Fig. 1 shows a photograph of the Star-of-FZK window mounting.

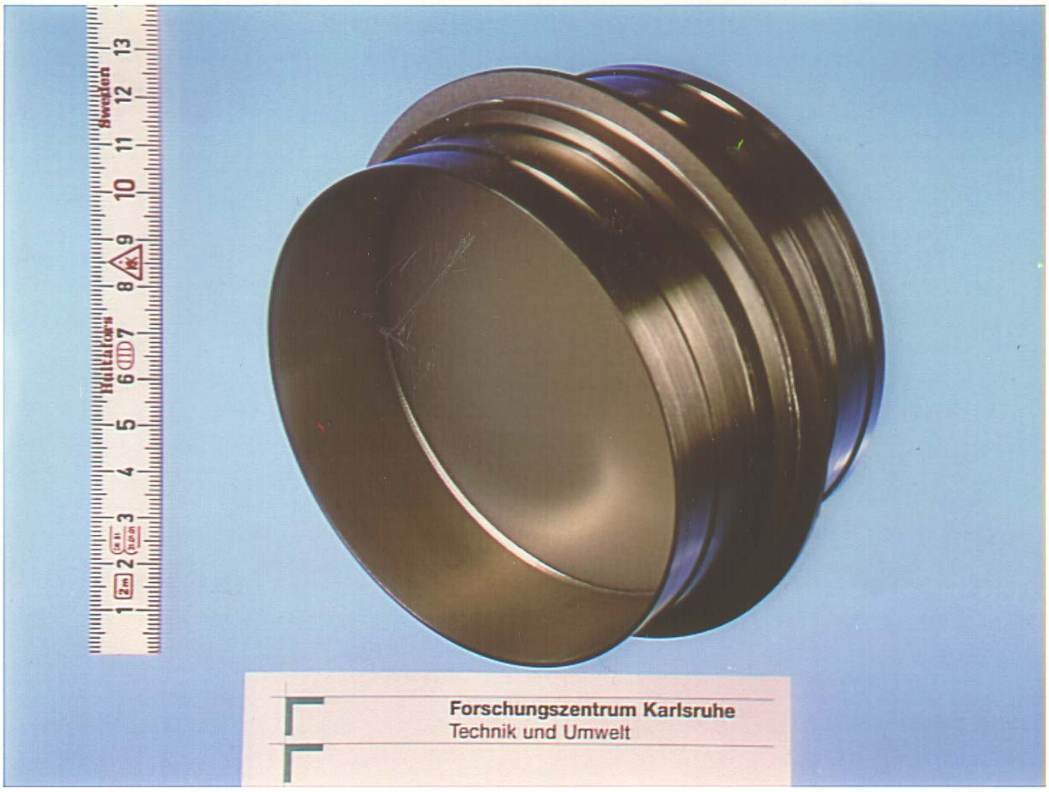


Fig. 1: Star-of-FZK window mounting with two Inconel cuffs and Molybdenum strengthening rings.

A new method of mounting MPACVD-diamond windows to metal flanges has been developed by DeBeers to allow vacuum bakeout to be performed at 550° C compared to the previous limit of 450° C. This increase in bakeout temperature promises dramatic reductions in the time taken to achieve optimum vacuum conditions in the gyrotrons. The new mounting utilizes a novel graded seal arrangement to achieve low thermal mismatch stresses and good dimensional stability during thermal cycling. A 75 mm diameter window mounted using this method has been shown to survive five thermal cycles up to 550° C for one hour without degradation of its sealing performance.

Finite element (FE) stress calculations performed at FZK for a typical mounting geometry also taking into account the brazing/bonding stress show that the maximum principal stress arises from the differential thermal expansion when bonding diamond to metal (205 MPa) and is always present. Copper, brazed at 800 °C, is chosen as metal (0.7 mm thick waveguide sleeves) because it is a very plastic and soft material (thermal expansion coefficient: α = 16.5 x 10^{-6} K^{-1} at 293 K). During a 0.5 MPa static overpressure event the maximum stress increases to 290 MPa and the transmission of 1 MW mm-power finally increases the maximum stress to 300 MPa. All these stress values are upper limits since a rigid connection between brazing collar and window disk was assumed. Test experiments performed by the JA Home Team at JAERI demonstrated that an RF-grade MPACVD-diamond window (aperture diameter = 70 mm, thickness = 2.25 mm) can withstand an overpressure of 1 MPa (0.04 mm displacement of center).

1.2 Status of high-power experiments

The results of high-power mm-wave experiments with diamond windows are summarized in Table 1. The pulse length is not limited by the MPACVD-diamond material but by outgassing inside the gyrotrons, arcing in the dummy loads or by the capabilities of the power supplies.

The design of a 1 MW, 170 GHz, CW, MPACVD-diamond window unit that fits to the Russian long pulse 170 GHz ITER prototype gyrotron was performed in collaboration of FZK and GYCOM (Fig. 2). The FZK window unit employed a single, edge cooled (water at e.g. 20° C) MPACVD-diamond disk (DeBeers, "Star of FZK") with an outer diameter of 119 mm, an aperture diameter of 100 mm and a thickness of 2.222 mm (6 $\lambda/2$). The outer rim for water

Table 1: Experimental parameters of high-power experiments (at 110-170 GHz) with MPACVD-diamond windows.

Material	Type	Power [kW]	Frequency [GHz]	Pulse Length [s]	Institution
diamond	single-disk water edge cooled	50	110	CW	CPI/FOM
		450	110	2.0	GYCOM-M/GA
		1050	110	5.0	CPI/GA
		550	110	10.0	CPI/GA
		1000	110	3.0	JAERI/TOSHIBA
		300	118	111.0	FZK/CEA/TED
		1000	140	10.0	FZK/CRPP/TED
		640	140	140	FZK/CRPP/TED
		470	140	180.0	FZK/CRPP/TED
		900	170	9.2	JAERI/TOSHIBA
		300	170	60.0	JAERI/TOSHIBA
		700	170	1.0	GYCOM-M/FZK

Note: **indicates that the power corresponds to that of a 0.8 MW HE_{11} mode.

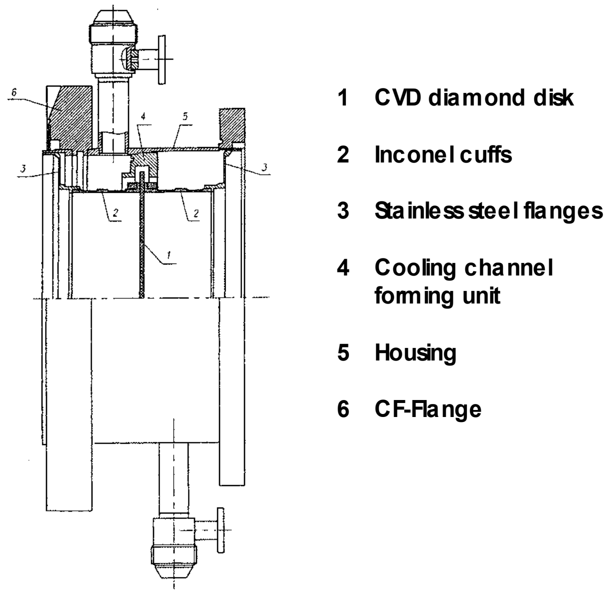


Fig. 2: 170 GHz ITER gyrotron output window with CVD diamond disk.

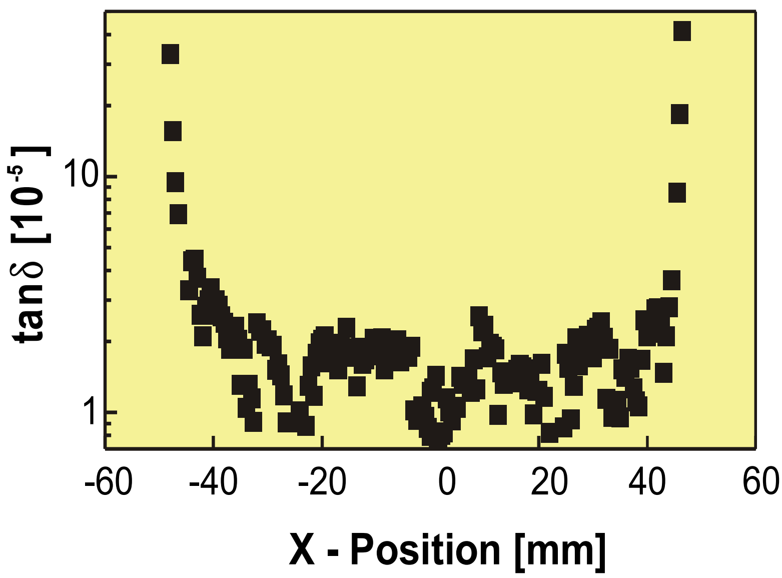


Fig. 3: Line scan of tanδ across the diameter of the "Star of FZK" window disk.

cooling is 5 mm wide. Thermal FE computations for a power of 1 MW at 170 GHz, a loss tangent of around $2 \cdot 10^{-5}$ (Fig. 3), a thermal conductivity of 1800 W/mK (at room temperature) and a heat transfer coefficient of 12 kW/m^2K to the cooling water (water flow: 13.5 l/min, water flow velocity: 2 m/s) show that the central window temperature will not be higher than approx. 64° C and the edge temperature is about 28° C. The absorbed power is lower than 530 W. Transmission of CW millimeter waves at power levels in excess of 1 MW should be possible. The simulations also show that steady state conditions are achieved in approximately 5 seconds. Nevertheless, on 9.11.1999 a failure took place after 20 routine pulses at this day (at this time the total amount of 0.7 MW pulses with pulse duration up to 1 s through the window was approximately 250). Two radial cracks from the window rim to the center are visible. The crack's lengths are 1 and 2 cm. The reasons for the window failure are still under discussion. These are: arcing, external mechanical stress (mounting of calorimeter), increased surface losses due to hydrogen-terminated diamond bonds or H^+ adsorption on ill-defined carbon surface states, or heating of the Inconel waveguide cuffs by stray radiation in the tube.

A new CVD-diamond disk has been purchased by the EU-Home Team and a new window unit (disk diameter 106 mm, thickness 1.852 mm, aperture 88 mm) has been manufactured in collaboration with the RF-Home Team and has been characterized at low power levels. During these measurements a sag of the window disk (120-140 µm) has been observed which corresponds to a static stress of approximately 0.7-0.8 MPa. Nevertheless, in order to fulfil this ITER task in time, high power tests have been performed. The gyrotron was operated at 1 MW output power and 1.5 s pulse length with 50% efficiency. On 26.05.2001 the window was broken after a pulse length of 0.2 s. The crack pattern is characteristic for failure induced by homogeneous and biaxial stress and thus reveals that the static stress due to the bow of the window was too large. A basic mechanical stress estimation which parameterises also the effective rigidity of the braze indicates that the tensile stress in the diamond disk at the brazed rim amounts to some 300 MPa. The fact, that the sag after the crack is even larger (200 μm) proves that it was introduced during the brazing to the waveguide cuffs.

A third CVD-diamond disk window unit does not show any sag (within the thickness and parallelism tolerances). The high power tests are scheduled for January 2002.

2. Report on the Test of a 1 MW, 170 GHz, CW CVD-Diamond Torus Window

2.1 Experimental setup

The CVD-diamond window disk (106 mm diameter, 1.852 mm thickness) was irradiated with a neutron fluence of $0.9 \cdot 10^{21}$ n/m^2. After characterization of the irradiated disk [5-8], two Inconel cuffs were brazed to the disk using an Al-braze (up to 450°C bakeout temperature). The CVD-diamond window unit of the EU-Hometeam was mounted into the test housing in a corrugated HE_{11} waveguide of the JA-Hometeam indicated in Figs. 4-6. It was intentionally designed to use it in a closed system with evacuated waveguides on both sides. Temperature monitoring of the disk is realized by six thermocouples, three of them mounted on the window edge (outer position) and three of them mounted near the inconel cuffs (inner position). The azimuthal position of the single thermocouples differ by 120 degrees for each set of thermocouples, the two sets are azimuthally shifted against each other by 60 degrees (indicated in Fig. 4).

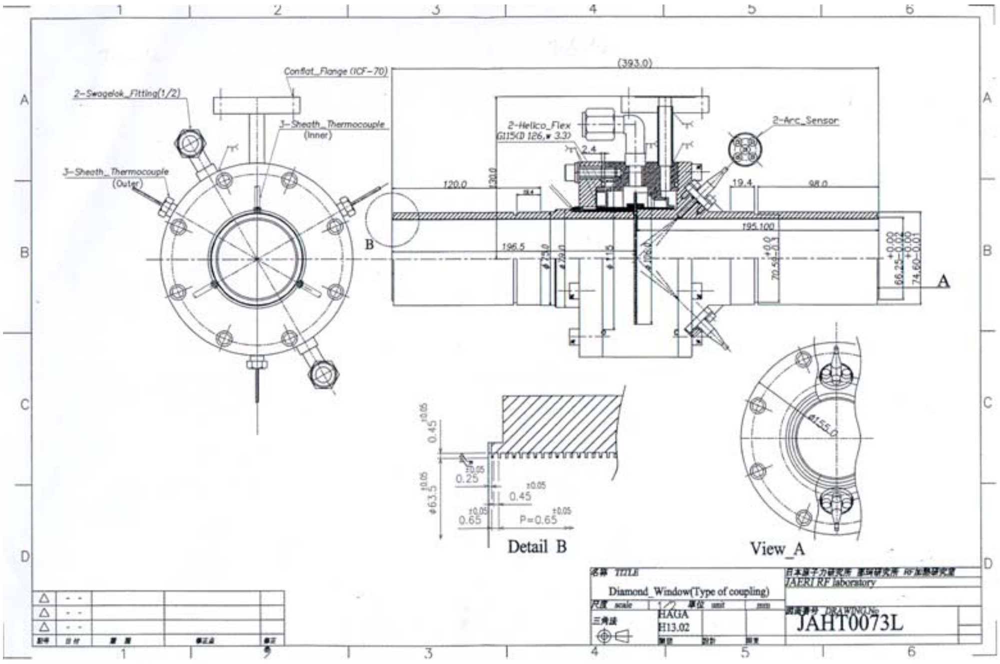
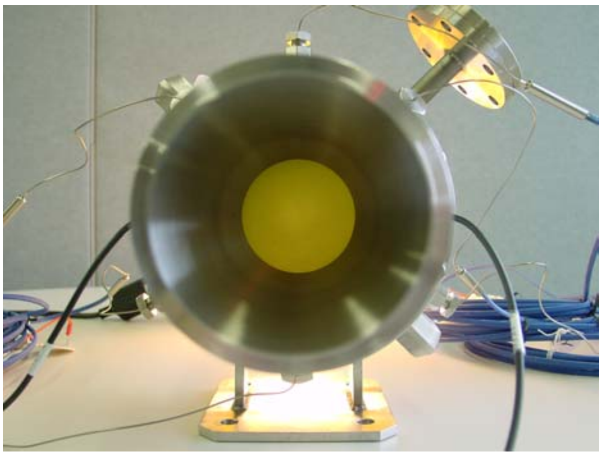


Fig. 4: Technical drawing of the housing for the irradiated CVD-diamond window disk.



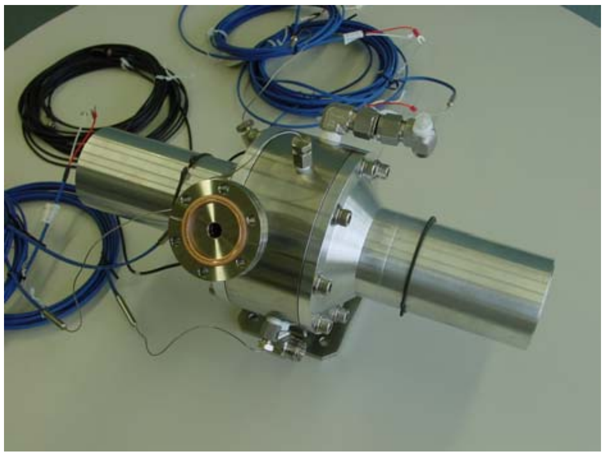
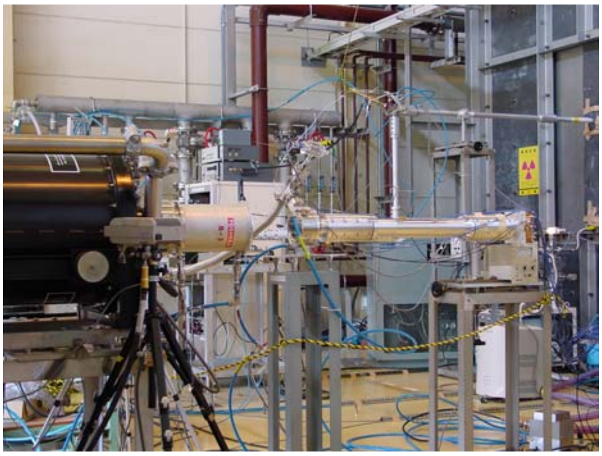


Fig. 5: View inside the HE_{11} wave-guides of the diamond window housing.

Fig. 6: Complete view of the diamond window housing.



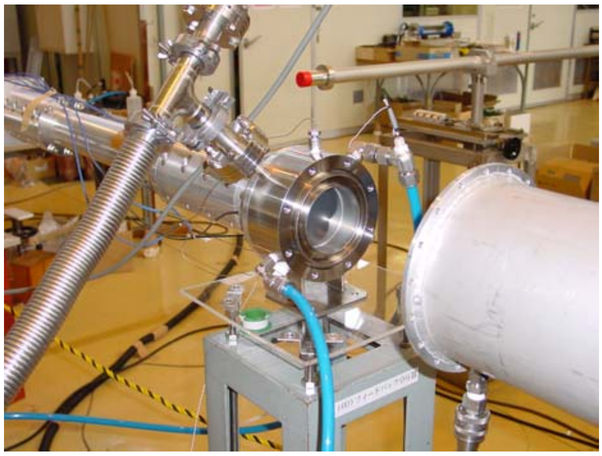


Fig. 7: Complete measurement set up including beam line, diamond window housing and CW-load.

Fig. 8: View of the built-in opened diamond window in front of the CW-load.

Arc monitoring of the window unit is realized by two arc sensors mounted on the gyrotron side of the housing (c.f. Fig. 4).

The microwave power supplied by the 170 GHz gyrotron was transmitted through a set of corrugated HE_{11} waveguides (63.5 mm inner diameter). At the date of the first measurement campaign in April 2001 the beam-line contained four miter bends and had a total length of approximately 8-10 meters.

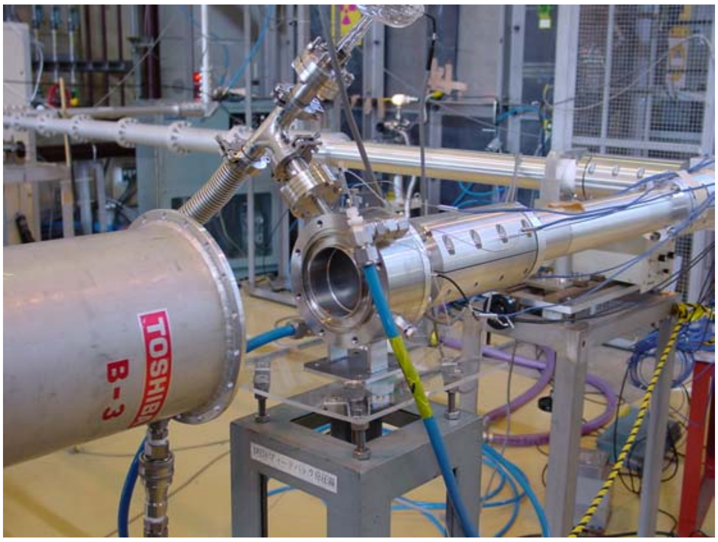


Fig. 9: Additional view of the transmission line, diamond window and load entry.

Instead of operation in a closed system with evacuated waveguides on both sides, the part on the load side of the window housing was removed and the microwave beam passed approximately 35 cm of air before it entered the load (c.f. Fig. 7-9). This made it possible to monitor visible effects and the temperature distribution on the window by a video-camera and a infrared (IR)-camera, respectively.

At the date of the second measurement campaign in August 2001 the number of miter bends in the beam-line was reduced to two and it had a total length of approximately 6-7 meters. Except for the first day the system was operated in the closed, evacuated state (c.f. Fig. 10).

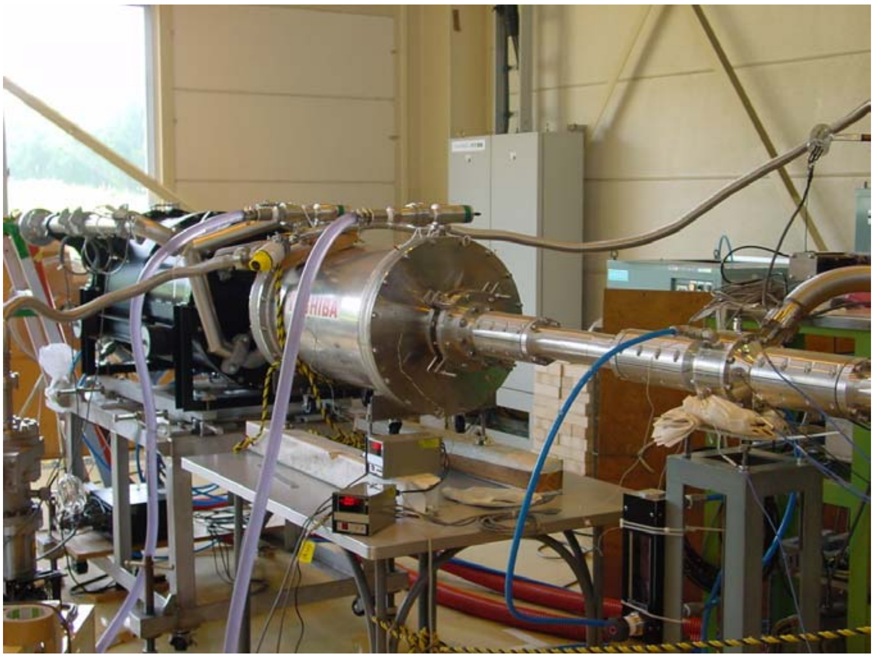


Fig. 10: Closed measurement setup including diamond window housing and load.

2.2 Measurements of April 2001

Due to technical problems the high power CW load supplied by Calabazas Creek Research (CCR) was not available at the beginning of the measurements. Instead a smaller evacuated Teflon-based load with a sapphire window was used which limited the beam power to 500 kW and the pulse length below approximately 100 ms.

The fact that the Teflon load used a sapphire window as vacuum barrier gave reason for beam reflections which caused the following problems: the beam pattern near the diamond window showed an asymmetric distribution, a significant temperature increase was monitored by the IR-camera at the flange near the diamond disk, also with an asymmetric shape. In addition the temperature monitoring by the thermocouples mounted on the disk showed this asymmetric behavior.

Attempts to suppress the load reflections by shifting the load window by approximately three degrees and by applying Ecosorb mats improved the situation but could not remove the problem in it's full dimension.

Nevertheless pulse lengths of 60 ms at a beam power of 500 kW were measured and gave reason for a temperature increase in the center of the uncooled CVD-diamond window of approximately 5°C. In addition a relatively small hot spot occurred near the center of the disk with a measured temperature increase of 20°C.

Maybe this hot spot was related to one of at least five bright visible spots (c.f. Fig. 11) that could be observed with the video-camera during the duration of the single pulses. These luminous spots were also observed by other research groups (like FZK/IHM and CPI) on (or behind) diamond windows of high power gyrotrons. Up to now the reason for this effect is unknown but an influence directly connected to the gyrotron (like x-rays from the collector) can be excluded.

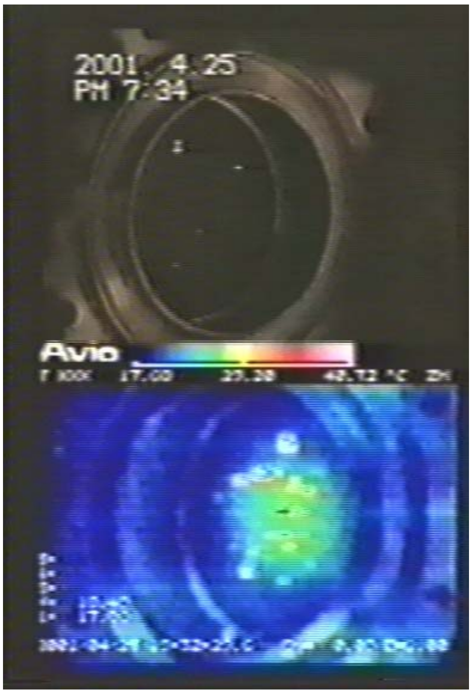


Fig. 11: Snapshots obtained with a video camera (upper part) and an IR-camera (lower part) during a 500 kW/300 ms pulse. Five visible spots can be observed.

Later the Teflon load was replaced by the CW CCR load (after its successful repair). Measurements of the beam pattern still showed a slightly asymmetric distribution but this was significantly better than the one obtained with the Teflon load.

First measurements at a power level of 500 kW and a pulse length up to 60 ms showed the same quantitative and qualitative behavior that was already observed the day before (luminous spots on the diamond disk, both visible with the IR- and video-camera). At a pulse length of 80ms the first arc in the CCR load occurred. After some conditioning the pulse length could be increased stepwise up to 350ms but spontaneous arcing in the load still occurred from time to time.

Fig. 12: The ECH-Group of the JA-Hometeam and Dr. S. Illy of the EU-Hometeam at JAERI.

The IR-camera measurements indicated a temperature increase of the uncooled window's center temperature of 19°C at a pulse length of 350 ms and a power level of 500 kW. The temperature of the hot spots indicated a temperature increase in these places up to 50°C. The temperature increases measured by the three thermocouples placed near the inconel cuff showed a slightly asymmetric behavior: the values were 2.0, 1.7 and 1.8 degrees, respectively.

With respect to the arcing in the CCR load at power levels of 500kW, the gyrotron output power was reduced to 300 kW on 26th of April. This made it possible to reach a pulse length of 2 s without arcing. The resulting temperature increase in the center of the CVD-diamond disk was 33° C (without window cooling). The three measured temperature increases at the inconel cuff were 14.0, 8.0 and 9.8°C, still showing the asymmetric behavior that may be caused by load reflections that heat up the flanches of the window housing.

After connecting the window unit with the cooling system the pulse lengths could be increased up to 3 s (still at 300 kW), resulting in a temperature increase in the disk's center of 30.5°C.

The operation was again limited by arcing in the load at a pulse length of 3.25 s.

After the first experiments with open geometry, the window unit was dismounted from the beamline to prepare the experiments with the closed evacuated setup.

The window surface was very clean both on the vacuum side and on the load side. There were no traces on the disk that may be connected to the bright luminous spots observed with the video-camera.

2.3 Measurements of August 2001

As mentioned above the number of miter bends in the experimental set-up of August 2001 was reduced to two, resulting in a shorter beam-line.

At the first day of the measurement campaign, the experiment was still performed in the open state, comparable to the experiments of April 2001. At a beam power of 500kW a maximum pulse length of 600ms could be reached, resulting in a temperature increase of approximately 40°C in the center of the uncooled CVD diamond disk. An increase of the pulse length was not possible due to arcing in the CCR load.

At the remaining days of the measurement campaign the experiment was operated in the closed, evacuated state (as indicated in Fig. 10). Since the evacuated load did not show any problems with arcing, a pulse length of 15s was possible at a transmitted beam power of 580kW (measured by the calorimetic load). More than 20 pulses were transmitted in the range of 500ms to 15s. The temperature increase measured at the inconel cuffs with three thermocouples was 27°C (for the 15s pulse), while the temperature increase at the disk edge was 9.0°C and 1.5°C. The large dispersion of the latter results may be due to bad connection of one of the thermocouples with the CVD-diamond disk. In addition, the measurement of the cooling water temperature showed an increase of 0.9°C after 15s.

Fig. 13 shows the dependence of the temperature increase of the disk at the inconel cuffs vs. pulse length, while Fig. 14 shows both the temperature increase at the disk edge (including the "bad" thermocouple) and the temperature increase of the cooling water vs. pulse length. Saturation of the cooling water temperature appears after a pulse length of 12s.

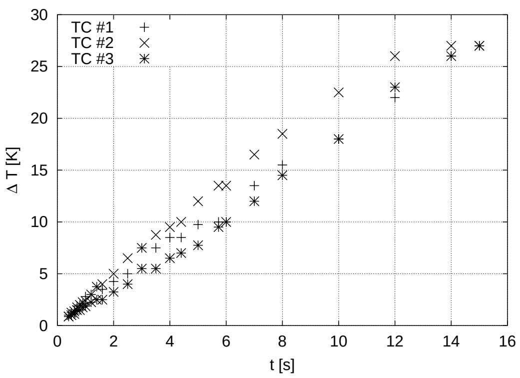


Fig. 13: Dependence of the temperature increase of the DVD-diamond disk at the inconel cuffs vs. pulse length.

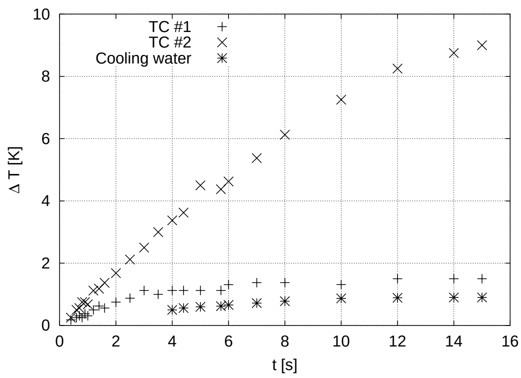


Fig. 14: Dependence of the temperature increase at the disk edge and of the cooling water vs. pulse length.

In addition longer pulses up to 132s were performed at a reduced beam power of 200kW. The resulting increase of the cooling water temperature was 0.29°C.

No arcing was observed in both cases (580kW and 200kW).

2.4 Comparison of finite difference calculations and measurements

Figure 15 shows a comparison of the measured temperature increases in the window center for the 500 kW/600 ms pulse and results obtained with a two-dimensional finite difference simulation for values of $\tan\delta = 4.8 \cdot 10^{-5}$, heat thermal conductivity k=1200 W/m·K and heat transfer coefficients at the disk edge and braze region of 50 W/m^2K and 4kW/m^2K (the uncooled case). This indicates an increase of the formerly measured thermal conductivity of 850 W/m·K [Ref. 5-7], maybe due to annealing effects.

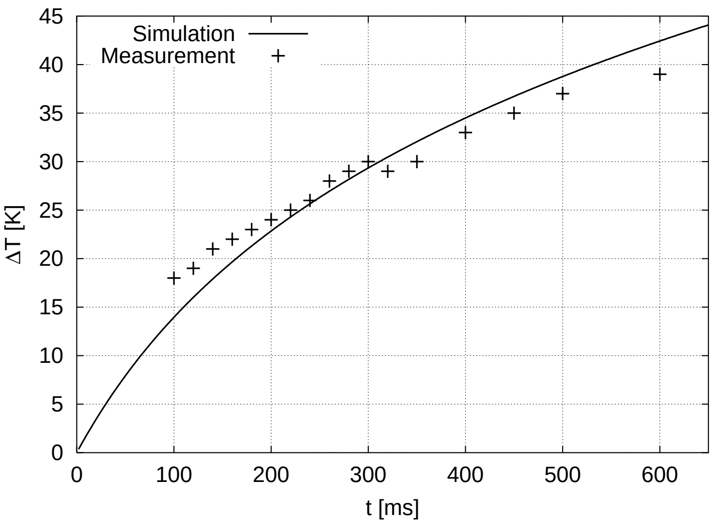


Fig. 15: Comparison of measured and calculated temperatures in the uncooled case.

2.5 CVD-diamond window experiments at CEA Cadarache

In October and November 2000 measurements with a CVD-diamond window unit in a closed evacuated transmission line were performed at CEA Cadarache. The diamond window "Super FZK" has an aperture of 80 mm (100 mm outer disk diameter), a thickness of 1.6 mm, a loss tangent of only $6 \cdot 10^{-6}$, a thermal conductivity of 2050 W/mK (at room temperature, but decreasing for increasing temperature) and has been bonded using Al-braze technique (c.f. FZKA report No. 6257, M. Thumm, O. Braz, R. Heidinger, R. Spörl, A. Arnod, P. Severloh, *ITER ECRF Coaxial Gyrotron and Window Development[ITER-Task No.: G52 TT 14 FE, ID-No.; GB7 – EU – T360]. Part II: Window Development – Final Report –*. FZK-ITP, FZK-IHM, Project Kernfusion, Association EURATOM-FZK). A schematical drawing of the complete experimental setup is shown in Fig. 16, a schematic drawing of the housing for the CVD-diamond window disk is indicated in Fig. 17.

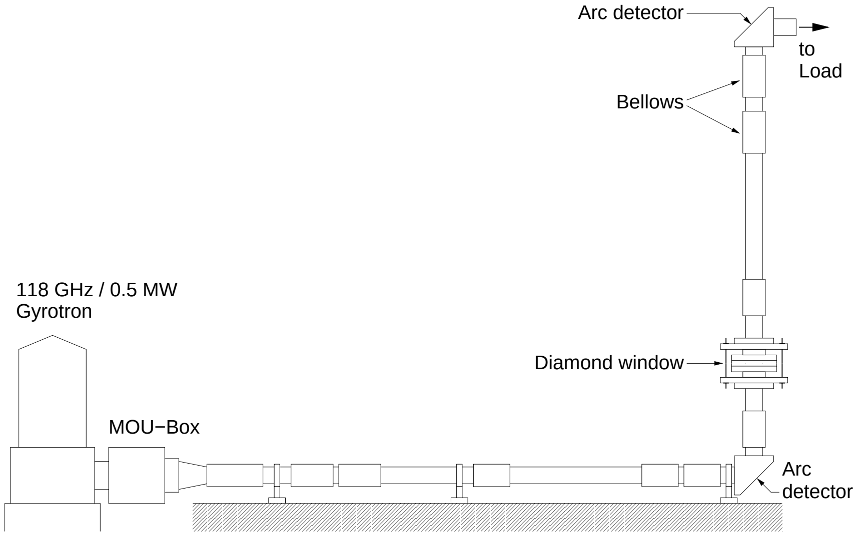


Fig. 16: Schematic drawing of the experimental setup at CEA Cadarache.

Due to technical problems with the 118 GHz gyrotron the requested output power of 500 kW could not be reached, instead a maximum output power of approximately 300 kW was available for a pulse length up to 111 s.

Since the CVD diamond window had a very low maximum tanδ of $6{\cdot}10^{-6}$, its expected theoretical power loss should not exceed 30 W at 300 kW beam power. The experimental values obtained with the window calorimeter differed significantly from this number, a power loss of 1.4 kW was measured.

A possible reason for this discrepancy is a non-negligible amount of high order modes created by mode conversion in the miter-bends and by reflected power from the load (at least 5%). Stray radiation from these modes with high Brillouin angle is able to penetrate efficiently both the 0.4 mm wide gaps between the corrugated waveguides and the CVD-diamond window disk as well as the gap represented by the diamond disk itself. The leaking stray radiations directly heats the cooling water of the window. During 5 s-shots in a well matched load the measured loss power was reduced to 0.9 MW.

After dismounting the diamond window unit, a large number of impurities was found on the disk, especially on the load side. These impurities were certainly caused by arcing due to the fact that the reflected power from the load was quite high.

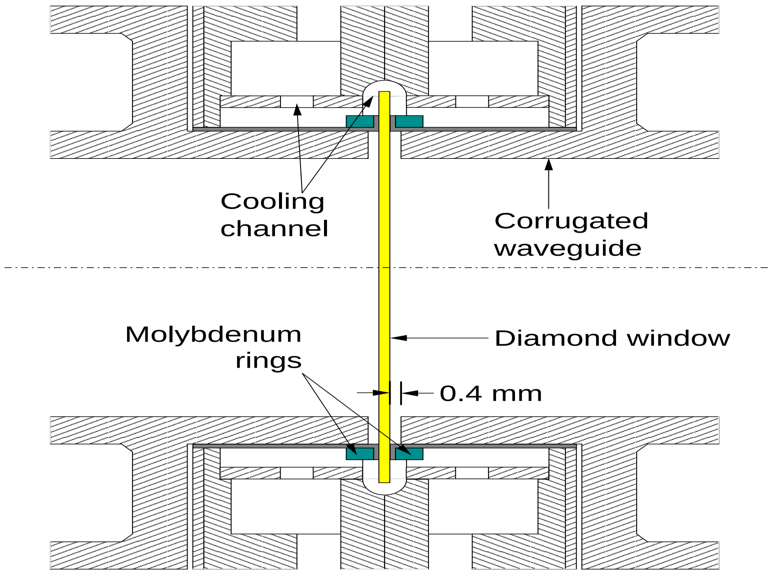


Fig. 17: Schematic drawing of the CVD diamond window housing.

3. Material Characterization of CVD Diamond for Megawatt ECH Windows

3.1 General aspects

The previous window development for ITER ECH clearly identified the Microwave Plasma Assisted (MPA-) approach of the Chemical Vapour Deposition (CVD) growth process of diamond as the presently uniquely suited method for growing large area diamond disks which fulfil the requirements of ultralow loss, outstandingly high thermal conductivity, and good mechanical strength [1]. The growth process implies a polycrystalline material which shows a pronounced variation of the lateral grain size along with disk axis. Principally the growth process starts with a fine-grained, defect rich structure at the interface to the substrate. As the process evolves preferential grain growth leads to coarsening of grains with lateral dimension exceeding 1/10 of the disk thickness. Depending on the actual control over the process parameters (such as plasma profile and substrate temperature), growth rates can vary over the area of the disk. For massive disks, often the appearance of micro-cracks in the grains and of non-diamond like phases at the boundaries and possibly also in the cracks are observed.

Inhomogeneities in the microstructure potentially cause local variations of the material properties which turn out to be most evident in dielectric loss. Generally large area disks are polished on the nucleation face (50-100 µm material for eliminating the starting layer) and on the growth face (generally several hundreds of micrometers of material removed to achieved flat surfaces). Depending on the actual surface quality considered, average roughness R_a ranges either around 100-150 nm ("standard polish", "lapped") or around 5 nm ("optical polish"). Only for an optical polish the disks show sufficiently low light scattering to identify local 'dark spots' which typically appear at eye-striking densities towards the edges of the disks as two dimensional structures which tend to form only after a certain thickness limit off the nucleation face (about 600 µm) and which generally extend over significant thickness ranges (100 – 400 µm). These structures are related with enhanced concentrations of non-diamond like phases identified by Raman spectroscopy. The concentration of these second phases goes along with the distribution of bulk losses in the specimen. The grain coarsening along the growth direction causes different values of mechanical strength depending on which face is submitted to tensile strength. These differences tend to narrow down with increased disk thickness. Disks in the window relevant range with thickness above about 1 mm apparently reach a rather flat thickness dependence, median values are found to tend towards 400 MPa for the growth face under tensile load, reaching about 600 MPa for the nucleation face.

CVD-diamond windows were investigated in the framework of the present task forming part of three major sets. Following various previous sets of smaller model disks produced by DeBeers (Charters, UK) referred as model disks ('evaluation', 'scale-up' and 'window grade') and the first large area, ultralow loss windows ('Star-of-FZK' and 'Super-FZK'), there was a set of initially four disks acquired from DeBeers which were later extended by a fifth one following the failure of the 'Star-of-FZK' window during high-power testing in collaboration with the RF Hometeam at Gycom (Moscow) (see chapter 1). This set was given the name '<u>prototype set</u>' and is referred to by internal codes 22DB1-22DB5. It includes the disks used for the 'ITER torus window demonstrator' (22DB2) (see chapter 2), for the 'Maquette 140 GHz W-7X gyrotron window' (22DB4) including the spare window (22DB3) and the replacement window for the 'Gycom ITER gyrotron demonstrator' (22DB5). The disk (22DB1) is now exchanged with CPI (Palo Alto, USA) for development of CVD-diamond windows with alternative brazing (Au-based). The second set is a <u>reference set</u> for large scale production amounting to 22 disks which are presently foreseen to be integrated into the W7-X ECH system. Actually the complete set of these disks plus three replacement disks, all with dimensions of 106 mm dia. x 1.8 mm thickness, was received and characterised for their mm-wave properties (internal codes ranging from 28DB1-49DB1). Finally there is the alternative ‘ellipsoidal reactor set’ produced by FhG-IAF (Freiburg, D) internally coded by a notation beginning with ‘ER’. Apart from demonstrating the potential of the growth process for achieving large area disks with comparable ultralow loss properties (ER4_69), possible effects of neutron irradiations on mechanical strength could be studied with disks cut from representative medium and large area sized disks having 62.5mm (ER6_55 and ER6_56) and 100 mm diameter (ER4_62). In addition two model disks have been acquired (ER4_71_1 and ER4_71_2) which show low loss and are particularly used to evaluate potential high temperature effects and to study the influence of different kinds of carbon layers with respect to surface losses.

3.2 Dielectric properties

The mapping of dielectric losses at 145 GHz for the bare disks of the prototype window set showed a significant lateral variation of dielectric losses at 145 GHz with median values of loss tangent (D50) ranging between 2.2×10^{-5} to 3.4×10^{-5}. Starting values of the distributions (D10) were typically close to 1.1×10^{-5} whereas terminal values (D90) reached up to $5\text{-}13 \times 10^{-5}$ [2]. Two disks (22DB1 and 22DB2) were investigated with a new high temperature set-up allowing mm-wave measurements at 145 GHz up to 500°C in vacuum. It was observed that the small temperature variation of the dielectric parameters, both ε_r and $\tan\delta$, which is known for temperatures below 350 K practically extends up to at least 700 K. The experiments showed, however, that additional thermal screening and control systems had to be introduced to relieve the existing uncertainties on the detailed temperature function for ε_r. As a consequence of this problem, $\tan\delta$ values were calculated in the data evaluation process assuming constant ε_r over the full experimental temperature range up to 800 K. In this approach, $\tan\delta$ increased by a factor of 2 between 300 K and 750 K. [3]. A revised version of the high temperature set-up was therefore realised which provides sufficient control over the thermal profiles to narrow down the systematic errors in ε_r to ± 0.01. By this way, it could be shown that there is a slight increase in ε_r and that the $\tan\delta$ dependence follows almost a flat line up to 650 K. The ‘Star-of-FZK’ window, which was measured with its braze removed after the failure event at Gycom, also followed a flat tanδ line (up to 650 K) comparable to those deduced for the disks from the prototype set (cf. Fig 18) [4].

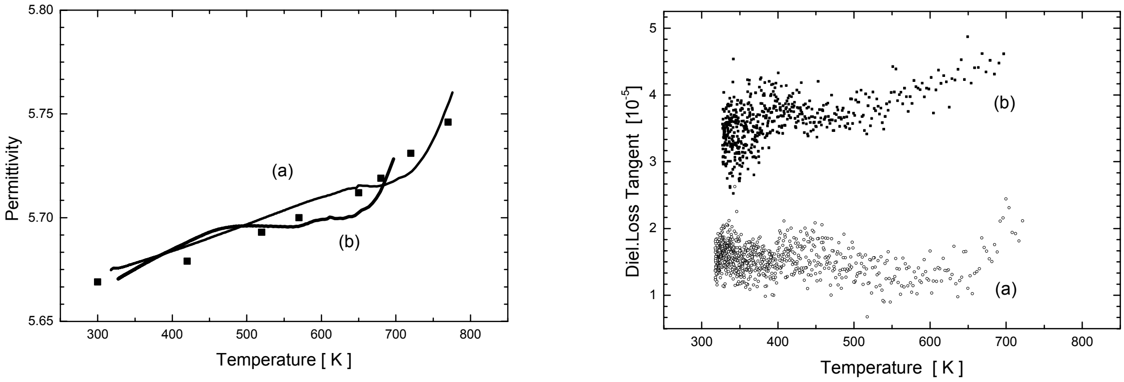


Fig. 18: The temperature dependence of the dielectric parameters at 145 GHz [4] for (a) a medium size model disk grown by the ellipsoidal reactor (ER4_71_1: 50 mm dia. x 1.05 mm)
(b) and the ‘Star-of-FZK’ disk (after removal from the brazed structure).

The disk 22DB2 was neutron-irradiated to $0.9 \cdot 10^{21}$ n/m^2 (E>0.1 MeV) to simulate the expected upper limit of structural damage (10^{-4} dpa) which can be tolerated for a primary window inside a EC wave transmission line [5,6]. At 145 GHz, the median values for tanδ (D50) were not significantly changed by the radiation effect[7,8], only after brazing at about 600-650°C into a metallic structure, D50 values increased from $3 \cdot 10^{-5}$ to $4 \cdot 10^{-5}$ (see Tab. 2) [9]. This can be explained by defect agglomeration during the brazing process. The window performance was inspected in high power transmission tests in collaboration with the JA Hometeam at JAERI (see chapter 2), the next step foreseen is the material characterisation (including thermal conductivity measurements) of the dismantled window.

Dielectric measurements on the reference set and the ellipsoidal reactor set have been performed at 90 – 100 GHz and at 145 GHz. It is found that typical disks have a median loss (D50) at 145 GHz of $2 \cdot 10^{-5}$, the distribution generally ranges from $1.5\text{-}2 \cdot 10^{-5}$ (D10) to $4 \cdot 10^{-5}$ (D90). Loss measured at 90 GHz gives an integral value for bulk absorption over a diameter of 10 mm which typically amounts to $2.5 \cdot 10^{-5}$ in good correspondence to values extrapolated by the empirical $f^{-0.5}$ scaling law. Results determined for the five of the more recently produced disks (39DB2,41DB1,42DB1,44DB1,45DB1) prove that process control has got still potential of refinement by which the lateral distribution and the median losses can be brought to the extremely low loss levels of tanδ ≈ $1 \cdot 10^{-5}$ which was demonstrated before for the special case of the 'Super-FZK' window [10] (cf. Tab. 2).

Tab. 2 Dielectric parameters determined for the reference set of high power CVD diamond windows.

				90 – 100 GHz			145GHz	
W7X Disk#	Internal code	Thickness (mechanical) [mm]	Thickn. (electr.) [mm]	εr	tanδ [10^{-5}]	D10 [10^{-5}]	tanδ 50% [10^{-5}]	90% [10^{-5}]
1	*28DB1*	1.806 - 1.810	1.811	5.672	2.5	0.9	1.7	3.7
2	*28DB2*	1.788 - 1.818	1.816	5.675	3.0	1.1	1.9	3.9
3	*29DB1*	1.792 - 1.797	1.817	5.672	2.4	<1	1.3	3.5
4	*31DB1*	1.795 - 1.797	1.806	5.672	3.0	2.7	3.7	6.1
4A	*31DB2*	1.786 - 1.806	1.793	5.669	1.6	1.0	1.4	2.7
5	*32DB1*	1.808 - 1.811	1.810	5.674	2.6	0.9	1.5	2.3
6	*33DB1*	1.796 - 1.810	1.799	5.670	2.5	0.9	1.3	3.3
7	*34DB1*	1.799 - 1.801	1.805	5.675	2.3	1.5	2.0	2.7
8	*35DB1*	1.798 - 1.806	1.806	5.673	2.8	1.6	2.3	3.6
9	*36DB1*	1.809 - 1.813	1.805	5.673	3.4	1.5	2.1	2.9
10	*37DB1*	1.805 - 1.808	1.813	5.669	4.0	2.3	2.9	4.0
10A	*37DB2*	1.789 - 1.802	1.790	5.670	1.6	1.3	2.3	4.1
11	*38DB1*	1.807 - 1.809	1.818	5.669	2.3	1.2	1.7	2.7
12	*39DB1*	1.798 - 1.804	1.806	5.667	2.2	2.2	2.8	4.3
12A	*39DB2*	1.796 - 1.806	1.803	5.670	1.2	1.2	1.6	2.7
13	*40DB1*	1.813 - 1.817	1.802	5.671	2.8	2.4	3.0	4.0
14	*41DB1*	1.805 - 1.811	1.806	5.672	1.5	1.2	1.4	1.7
15	*42DB1*	1.809 - 1.818	1.813	5.670	1.7	1.0	1.4	1.7
16	*43DB1*	1.806 - 1.817	1.805	5.669	2.5	2.1	2.6	3.7
17	*44DB1*	1.790 - 1.800	1.782	5.670	1.4	1.2	1.7	2.5
18	*45DB1*	1.794 - 1.805	1.798	5.671	1.1	1.0	1.5	3.7
19	*46DB1*	1.790 - 1.806	1.801	5.670	1.3	1.5	2.5	5.1
20	*47DB1*	1.786 - 1.800	1.796	5.672	2.2	2.3	2.8	4.1
21	*48DB1*	1.803 - 1.808	1.806	5.669	1.9	1.8	2.7	4.4
22	*49DB1*	1.788 – 1.800	1.799	5.670	1.8	1.8	2.5	3.9
Torus 1	*22DB2*	Pre n-irrad.	1.864	5.673	2.8	1.2	3.4	7.6
Torus 1	*22DB2*	Post n-irrad.	1.863	5.677	4.2	1.8	2.8	4.6
Torus 1	*22DB2*	Post n/ Brazed	1.867	5.650	4.9	3.7	4.1	4.6

Dielectric losses in CVD diamond were identified to be potentially composed of normal 'bulk losses', which form the only term in bare disks, and of surface losses, which incidentally show up in brazed components. The first example was documented for the 'Star-of-FZK' window which created a special concern because of its failure which occurred for still unknown reasons. Measurements were therefore specially focused to a dedicated variation of surface sensitivity in the hemi-spherical Fabry-Perot resonator technique used. This was achieved by inspecting the apparent dielectric loss as a function of the number of half-wavelength measured between 90-100 GHz. A systematic increase of the apparent loss tangent is obtained when changing by frequency steps from the condition of 'resonant thickness' (providing bulk losses only) to the condition of 'anti-resonant thickness'; the apparent losses obtained in this case is describes with fair precision the effective absorption level which includes the surface terms. In principle three characteristic origins of surface losses were assigned, arising from fully hydrogenated diamond surfaces (potential loss enhancement up to factor 100), graphitised surface (potential loss enhancement up to factor 10) and ill-defined surfaces (potential loss enhancement up to factor 4). For Al-brazed windows the case of ill-defined surface appears to be most relevant and for Au-brazed windows, graphitised surfaces form a potential problem [11]. Mechanical and chemical cleaning procedures are under development to reduce these effects (cf. Fig. 19a) [12], as safe solution surface polishing possibly combined with surface coating are being studied.

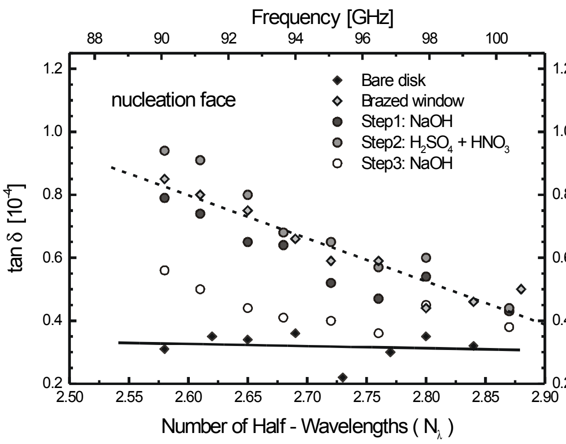


Fig.19a: Apparent dielectric loss values measured at 90-100 GHz at different processing stages and chemical treatments of the spare Maquette window (22DB3).

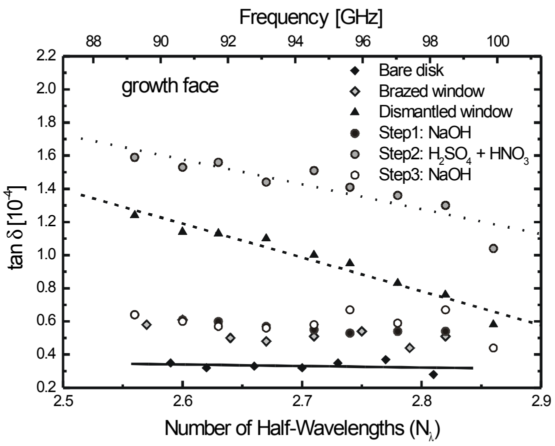


Fig.19b: Apparent dielectric loss values measured at 90-100 GHz at different testing stages of the Maquette window (22DB4).

Another issue of concern is emission of light localised at small individual spots over the CVD-diamond window installed in the 140 GHz Maquette high power tube for W7-X at FZK. Comparable effects could be observed with the Torus demonstrator window (chapter 2, Fig. 11). Optical and infrared inspection has been performed in order elucidate the origin of this effect. As an important result, it could be excluded that hot spots form in the non-diamond like phases which are the source of the effective bulk losses. For it was demonstrated, that the spare brazed Maquette window (22DB3) did not show the light emission when it was introduced as a transmission window in the high power quasi-optical beam line at a position of similar power density as for the Maquette tube output window [13]. Present experimental evidence tends to favour the interpretation that deposits, which are found at various windows after high power transmission tests, are stabilised under the vacuum conditions of the tube and allow for still unidentified light emission processes [14].

The studies on the dismantled Maquette window show that there were surface loss terms present which did not exist in the brazed structure before high power test. Indeed, acid and hydroxide treatments had been applied on the gyrotron window between different phases of high power irradiation to learn whether the light emission was related with an existing surface adsorbate or contamination. While there was no reduction in the density of light emitting spots seen, clearly the surface losses were enhanced probably by insufficient hydroxide treatment to remove the protonic surface states induced by the acid treatment (cf. Fig. 19b). The surface losses could be effectively removed by hydroxide treatment of the dismantled disk. Also the results of the loss mapping available from different stages of the window integration show that there was no substantial change in the bulk losses under high power operation (cf. Fig. 20).

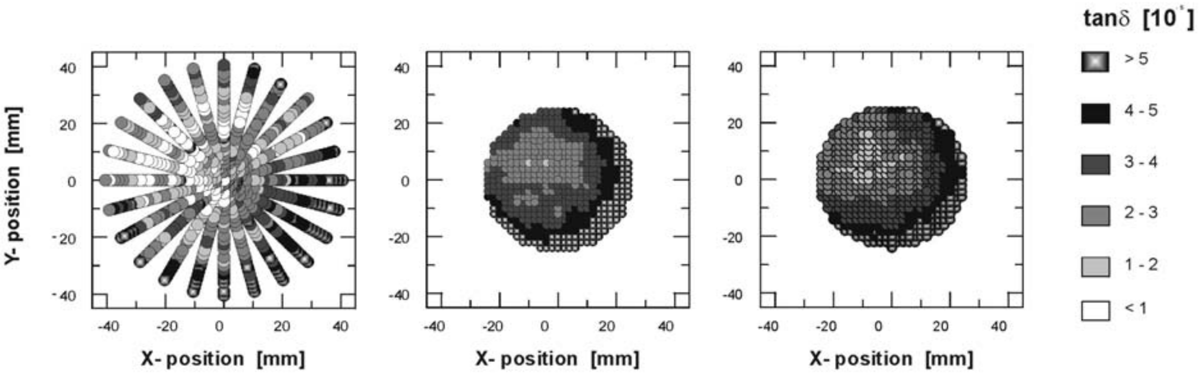


Fig. 20: The distribution of bulk losses measured at 140 GHz at different stages of the Maquette window test
Left: Bare disk ($D50=2.6\cdot10^{-5}$), Centre: Dismantled disk ($D50=3.4\cdot10^{-5}$), Right: Dismantled disk after hydroxide cleaning ($D50=3.3\cdot10^{-5}$).

3.3 Thermal and mechanical properties

Thermal conductivity measurements were performed for the torus demonstrator window (22DB2) and compared to results of model disks (30mm and 40 mm dia.). Thermal conductivity for the torus demonstrator window in the pre-brazed condition was found to be strongly reduced (840 W/m·K as compared to 1800-2000 W/m·K for unirradiated CVD diamond). These findings are in agreement with the physical model proposed before which identifies single point defects as the predominant factor affecting the thermal conductivity [8]. Potential partial recovery by defect agglomeration during the brazing process could not be investigated with the given test method (photoacoustic set-up) as it was found that without coating by graphite no satisfactory deposition of the laser energy was achieved for detecting the thermal response of the specimen. It was decided not to apply the graphite coating because the risk of additional surface losses for the high power tests at JAERI was to too serious.

Mechanical strength tests were performed with specimens (11 mm dia.) cut from medium size diamond disks (62 mm dia.) from the ellipsoidal reactor set. The mechanical tests were performed with a fixed specimen orientation relative to the diamond growth (growth face under tension) to account for intrinsic anisotropies in grain size. The neutron irradiated disks (fluence: 10^{21} n/m^2, E>0.1MeV) from the Diamond windows (ER4_62, ER6_55, and ER6_56) all group between 400 MPa and 440 MPa (cf. Fig. 21). Even though the various

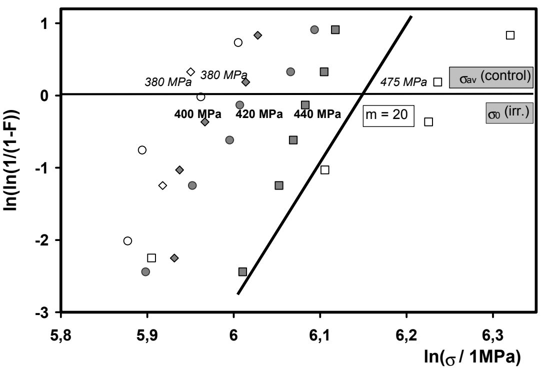


Fig. 21: Weibull analysis of the critical strength measured in the three specimen sets of CVD diamond after neutron irradiation to 10^{21} n/m^2 (E>0.1MeV) (full symbols): ER4_62(squares), ER6_55(circles) and ER6_56(diamonds). Inserted numbers indicate the median strength values. The straight line represents a line with a slope given for Weibull modulus of m=20.). For the unirradiated control specimens (open symbols), only the mean value of the measured strength data are indicated due to limited specimen numbers.

model windows covered a significantly wide thickness range (0.9 – 1.5 mm), no systematic thickness dependence was found. Such dependence could be expected from the grain coarsening effect put to evidence for these CVD diamond grades (mainly for disks of much smaller thickness). The finding is especially obvious for the results obtained for the 4” window (ER4_62) where the median value (440 MPa) is clearly larger than the one observed for window ER6_55 (400 MPa) despite of a similar even somewhat smaller thickness. The values obtained agree well with values extrapolated into this range from earlier studies of unirradiated diamond disks which imply a range of 370 – 450 MPa. Also the results obtained with few unirradiated control specimens confirm the absence of radiation induced degradation. The apparent larger strength values in the unirradiated control specimens of the 4” window are not judged to contradict to this view. Rather these data are interpreted as an indirect evidence for particularly grown-in stresses during the growth of this particular window [15].

The structural damage introduced by fast neutrons at a 10^{-4} dpa level is apparently of minor relevance to the mechanical strength in CVD diamond [8]. This goes clearly along with expectations that could be justified by previous findings for sapphire and alumina ceramics where mechanical strength degradation sets in at above 10^{-1} dpa.

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