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FZKA 6257

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Part II: Window Development

– Final Report –

M. Thumm, O. Braz, R. Heidinger, R. Spörl, A. Arnold, P. Severloh

Institut für Technische Physik
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ITER ECRF COAXIAL GYROTRON AND WINDOW DEVELOPMENT

Part II: Window Development

- Final Report -

Task No.: G52 TT 14 FE
ID-No.: GB7 - EU – T360

Executive Summary:

Two metallization/bonding techniques for CVD-diamond disks have been developed in collaboration with DeBeers. The disadvantage of the first method which uses Al-based braze (GB6-EU-T245/6) is the relatively low allowed bakeout temperature of the window unit of 450°C (guaranteed by DeBeers). Therefore, further tests have been performed on metallized disks which have been bonded with Au-based brazing on both sides to Inconel 600 cylinders to form part of a full window assembly (outer disk diameter = 50 mm, disk thickness = 1.8 mm, window aperture = 40 mm). The Inconel cylinders are strengthened by Molybdenum rings in order to reduce thermal expansion during bakeout. These sub-assemblies have been subjected to the bakeout cycle of a gyrotron up to 550°C with the result that the bonding at both sides got a leak. This shows that the Au-based braze is not as elastic as the former Al-based braze. Thus, the next bonding/brazing tests will be performed either with Molybdenum (small thermal expansion) or with Copper (elastic) waveguide sleeves.

The design of a 118 GHz, 0.5 MW, 210 s CVD-diamond window was finished and the window unit has been manufactured at TTE. The universal design allows tests in an evacuated HE_{11}-transmission line at CEA Cadarache and also direct mounting to a 118 GHz TTE-gyrotron. The window has an aperture of 80 mm (100 mm outer disk diameter), a thickness of 1.6 mm, a loss tangent of only $6 \cdot 10^{-6}$ (world record!), a thermal conductivity of 2050 W/mK (at room temperature) and has been bonded using the Al-braze technique. The window will be tested in Spring 1999 at CEA Cadarache when the long-pulse 118 GHz gyrotron will be available.

In collaboration with the JA Home Team high-power tests on a 170 GHz gyrotron equipped with a CVD-diamond window were performed. The window aperture is 83 mm. The polycrystalline diamond disk has a diameter of 96 mm, a thickness of 2.23 mm, a loss tangent of $1.3 \cdot 10^{-4}$, a thermal conductivity of 1800 W/mK (at room temperature) and was also bonded with the Al-braze technique. The tests were very successful (450 kW, 8s: 3.6 MJ). The maximum window center and edge temperatures were measured to be 150°C and 13°C, respectively, with a saturation time of approximately 5 s.

Three CVD-diamond disks with 106 mm outer diameter and 1.8 mm thickness have been ordered at DeBeers for the development of a 140 GHz, 1 MW, CW prototype gyrotron for W7-X with single-stage depressed collector (efficiency > 45 %).

Following previous ITER work on ECRF gyrotron window development (see GB6-EU-T245/6), which demonstrated the availability and the up-scaling potential of CVD-diamond grades with low dielectric losses, neutron irradiation tests were extended to fluences of 10^{21} n/m^2 (E > 0.1MeV). Even at this damage level (10^{-4} dpa) which corresponds to the recommended upper fluence level for cryogenically-cooled Sapphire windows, no critical radiation enhanced losses were observed at 90 GHz and 145 GHz (see also GB7 – EU – T246). Therefore the preparation for forthcoming irradiation tests of actual ITER test window component material was set to this fluence level. The unsettled issue of degradation of thermal conductivity at 10^{-5} dpa damage level was the reason for a pending special neutron irradiation at 10^{-4} dpa of specimens for thermal conductivity measurements.

In the material characterisation for actual CVD-diamond window materials, several disks of white grade material with diameters of 100 – 119 mm and thickness between 1.6 mm and 2.3 mm were investigated which were produced at DeBeers. In the latest disks, internally named "SUPER-FZK" (100 mm dia. x 1.60 mm) and "STAR of FZK" (119 mm x 2.25 mm), the center showed low losses which were nearly constant between 70 K and 370 K just like the permittivity of 5.67. Inhomogeneities in mm-wave properties which were important in earlier disks were checked in terms of variations in the surface contributions from the nucleation and the growth face as well as in terms of distribution of losses across the area of the disks. The recent two large disks which had the starting layer removed from the nucleation side did not show any significant differences in the losses between the nucleation and the growth side. Over a mapped inner area of 70 mm diameter losses were found at 145 GHz not exceeding $1 \cdot 10^{-5}$ for the "SUPER-FZK" disk and not exceeding $2 \cdot 10^{-5}$ for the "STAR of FZK" disk. The window material development was supported by dielectric property measurements of grades from potential alternative European and American sources. A first set of 12 mm diameter disks for mechanical strength tests were received from corresponding white grade material. The typical values for the 0.26 mm thick disks of 700 MPa for the growth side under tension fit well into a relationship established for strength data observed from another source studied in the framework of a separate CVD-diamond development task. The apparent correlation of ultimate bending strength with diamond grain size implies strength data as high as 2000 MPa to be expected for the nucleation side under tension. Compared to standard technical ceramics (such as alumina), CVD-diamond of a given quality has a remarkably high Weibull modulus (m > 20), i.e. very small distribution of the strength values.

KOAXIALGYROTRON- UND FENSTER-ENTWICKLUNG FÜR ITER ECRF

Teil II: Fenster-Entwicklung

- Schlußbericht -

Task No.: G52 TT 14 FE
ID-No.: GB7-EU-T360

Kurzfassung:

In Zusammenarbeit mit der Fa. DeBeers wurden zwei Metallisierungs- und Löttechniken für CVD-Diamant entwickelt. Der Nachteil der ersten Methode, die ein auf Aluminium basierendes Lot verwendet, ist die relativ niedrige erlaubte maximale Ausheiztemperatur der Fenstereinheit von 450°C (garantiert von DeBeers). Deshalb sind weitere Tests mit metallisierten Scheiben durchgeführt worden, die mittels eines auf Gold basierenden Lotes auf beiden Seiten mit Inconel-600-Zylindern verbunden wurden, um so eine vollständige Fensteranordnung zu erhalten (Scheibenaußendurchmesser = 50 mm, Scheibendicke = 1,8 mm, Fensterapertur = 40 mm). Die Inconelzylinder werden durch jeweils einen Molybdänring verstärkt, um die thermische Ausdehnung während der Ausheizung zu verringern. Diese Testfenster wurden demselben Ausheizzyklus bis zu 550 °C, wie es bei Gyrotrons üblich ist, unterzogen mit dem Resultat, daß die Lötverbindungen auf beiden Seiten Vakuumlecks aufwiesen. Dies zeigt, daß das auf Gold basierende Lot nicht so elastisch wie das frühere auf Aluminium basierende Lot ist. Die nächsten Metallisierungs- und Verbindungstests werden daher entweder mit Molybdän (geringe thermische Ausdehnung) oder mit Kupfer (elastisch) – Hohlleiterzylinder durchgeführt.

Die Auslegung eines 118 GHz-0,5 MW-210s–CVD-Diamantfensters wurde abgeschlossen und die Fenstereinheit bei TTE hergestellt. Der universelle Entwurf erlaubt Tests in einer evakuierten HE_{11}-Übertragungsleitung bei CEA-Cadarache und ebenso die direkte Montage an ein 118 GHz-TTE-Gyrotron. Das Fenster besitzt eine Apertur von 80 mm (Scheibenaußendurchmesser = 100 mm), eine Dicke von 1,6 mm, einen Verlusttangens von $6 \cdot 10^{-6}$ (Weltrekord!), eine Wärmeleitfähigkeit von 2050 W/mK (bei Zimmertemperatur) und wurde mit der Al-Löttechnik gelötet. Das Fenster soll im Frühjahr 1999 bei CEA-Cadarache getestet werden, wenn das Langpuls-118 GHz-Gyrotron verfügbar ist.

In Zusammenarbeit mit dem JA Home Team wurden mit einem 170 GHz-Gyrotron Hochleistungstests eines in die Röhre eingebauten CVD-Diamantfensters durchgeführt. Die Fensterapertur ist 83 mm. Die polykristalline Diamantscheibe hat einen Durchmesser von 96 mm, eine Dicke von 2,23 mm, einen Verlusttangens von $1,3 \cdot 10^{-4}$, eine Wärmeleitfähigkeit von 1800 W/mK (bei Zimmertemperatur) und wurde ebenfalls mit der Al-Löttechnik gelötet. Die Tests waren sehr erfolgreich (450 kW, 8 s: 3,6 MJ). Die maximale Zentrums- bzw. Randtemperatur wurde zu 150°C bzw. 13°C gemessen, mit einer Sättigungszeitkonstanten von ungefähr 5 s.

Für die Entwicklung eines 140 GHz-1 MW-CW-Prototyp-Gyrotrons mit einstufigem Kollektor mit Gegenpotential (Wirkungsgrad > 45 %) für W7-X wurden drei CVD-Diamantscheiben mit 106 mm Außendurchmesser und 1,8 mm Dicke bei DeBeers bestellt.

Es wurden Bestrahlungstests mit Neutronen bei einer erhöhten Fluenz von 10^{21} n/m² (E> 0,1 MW) durchgeführt. Selbst bei dieser Bestrahlungsstärke (10^{-4} dpa), die der für kryogengekühlte Saphirfenster empfohlenen oberen Fluenzgrenze entspricht, wurden bei 90 GHz und 145 GHz keine kritischen, durch Strahlung reduzierte erhöhten Verluste gemessen. Daher wurden Vorbereitungen getroffen, zukünftiges Scheibenmaterial für ITER-Testfenster bei dieser Bestrahlungsstärke zu testen. Die noch nicht geklärte Verschlechterung der Wärmeleitfähigkeit bei 10^{-5} dpa Zerstörungsniveau macht eine weitere Neutronenbestrahlung, bei 10^{-4} dpa für Wärmeleitfähigkeitsmessungen erforderlich.

Zur Materialcharakterisierung von aktuellen CVD-Diamantfenstermaterialien wurden mehrere von DeBeers produzierte Scheiben aus weißem Material mit Durchmessern von 100-119 mm und Dicken zwischen 1,6 mm und 2,3 mm untersucht. Die zuletzt gelieferten Scheiben, intern mit "SUPER-FZK" (100 mm x 1,60 mm) und "STAR of FZK" (119 mm x 2,25 mm) bezeichnet, zeigen im Zentrum niedrige Verluste, die, ebenso wie die Permittivität von 5,67, im Bereich von 70 K bis 370 K nahezu konstant sind. Inhomogenitäten der mm-Welleneigenschaften, die bei früheren Scheiben wesentlich waren, wurden durch Variationen der Beiträge der Nukleations- und Aufwachsflächen und durch Vermessung der Verlustverteilung über die Scheibenfläche untersucht. Die letzten zwei großen Scheiben, bei denen die erste Aufwachsschicht auf der Nukleationsseite abgetragen wurde, zeigten keine signifikanten Unterschiede der Verluste auf der Nukleations- bzw. der Wachstumsseite. Über eine vermessene Fläche von 70 mm Durchmesser sind die Verluste bei 145 GHz nicht größer als $1 \cdot 10^{-5}$ bei der "SUPER-FZK"-Scheibe und $2 \cdot 10^{-5}$ bei der "STAR of FZK"-Scheibe. Diese Entwicklung von Fenstermaterialien wurde auch durch Untersuchungen der dielektrischen Eigenschaften von Testscheiben von alternativen europäischen und amerikanischen Quellen unterstützt. Ein erster Satz von Diamantscheiben eines entsprechenden weißen Materials mit 12 mm Durchmesser für Tests der mechanischen Stärke wurde geliefert. Die typischen Werte von 700 MPa für die 0,26 mm dicken Scheiben im Falle, daß die Wachstumsseite unter Zugspannung steht, passen gut zu den in einem anderen CVD-Diamant-Entwicklungsprogramm beobachteten Daten. Die augenscheinliche Korrelation der Bruchfestigkeit mit der Diamantkorngröße läßt im Falle, daß die Nukleationsseite unter Zugspannung steht, Festigkeitswerte in der Gegend von 2000 MPa erwarten. Verglichen mit Standard-Strukturkeramiken (wie z.B. Aluminumoxid), hat CVD-Diamant einen beachtlich höheren Weibull Modul (m>20), d.h. eine sehr enge Verteilung der mechanischen Spannungswerte.

Contents

1. Development of Metallization/Bonding Techniques for a Diamond Window (Subtask 2.1)

In order to integrate microwave plasma assisted chemical vapour deposition (CVD)-diamond disks in ultra-high vacuum tight window assemblies for gyrotrons and plasma torus, the problems of metallization and brazing to cylindrical metal sleeves for welding to the window mounting have to be solved. The bakeout temperature of gyrotrons at the factory site is between 450°C and 550°C, depending on the company. The brazing has to withstand these temperatures. Recent data on relative thermal expansion of common microwave window and ferrule materials are shown in Fig. 1. The temperature dependence of the thermal expansion coefficient α of CVD diamond is plotted in Fig. 2.

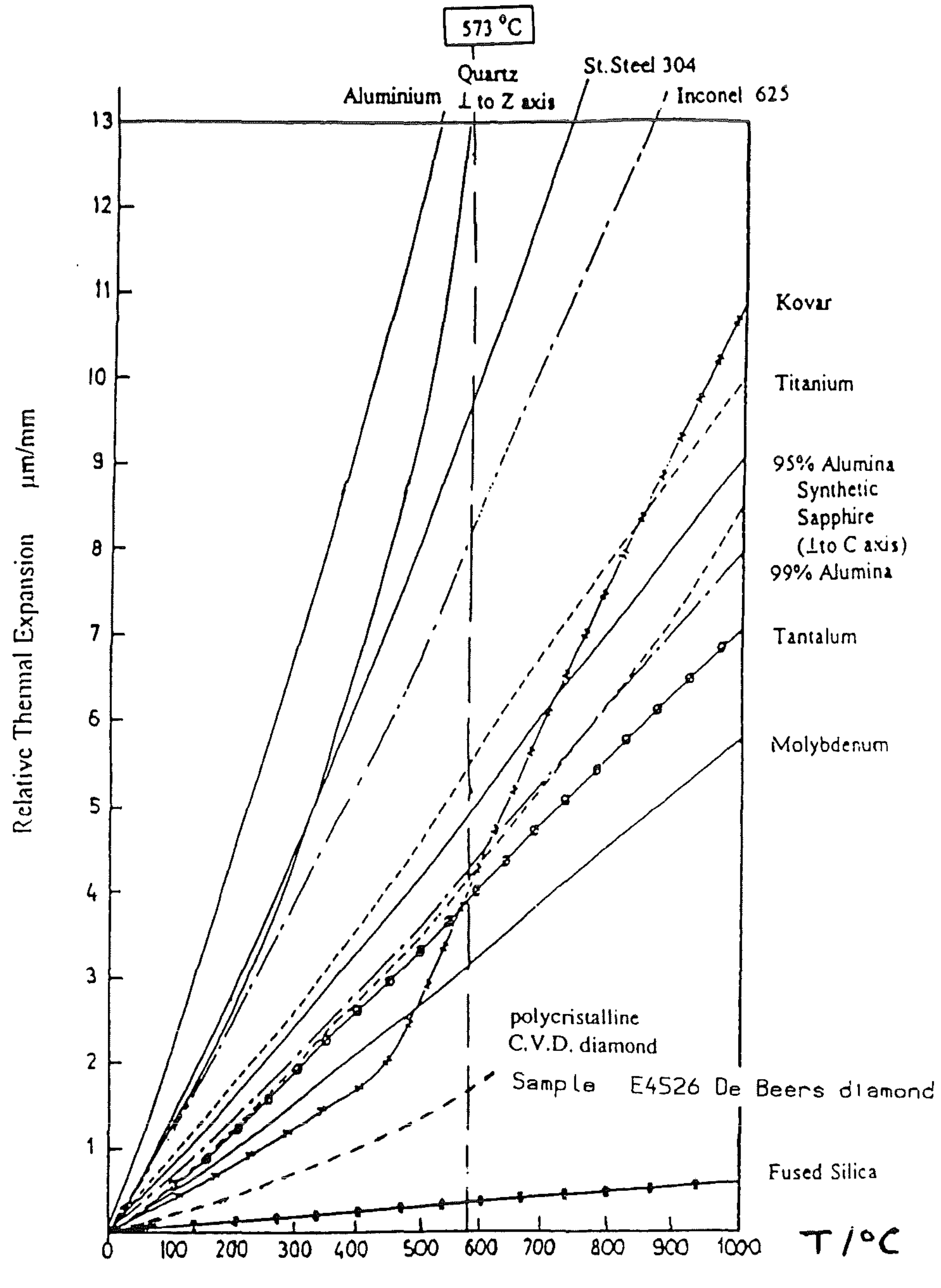


Fig. 1: Relative thermal expansion of common window and ferrule materials.

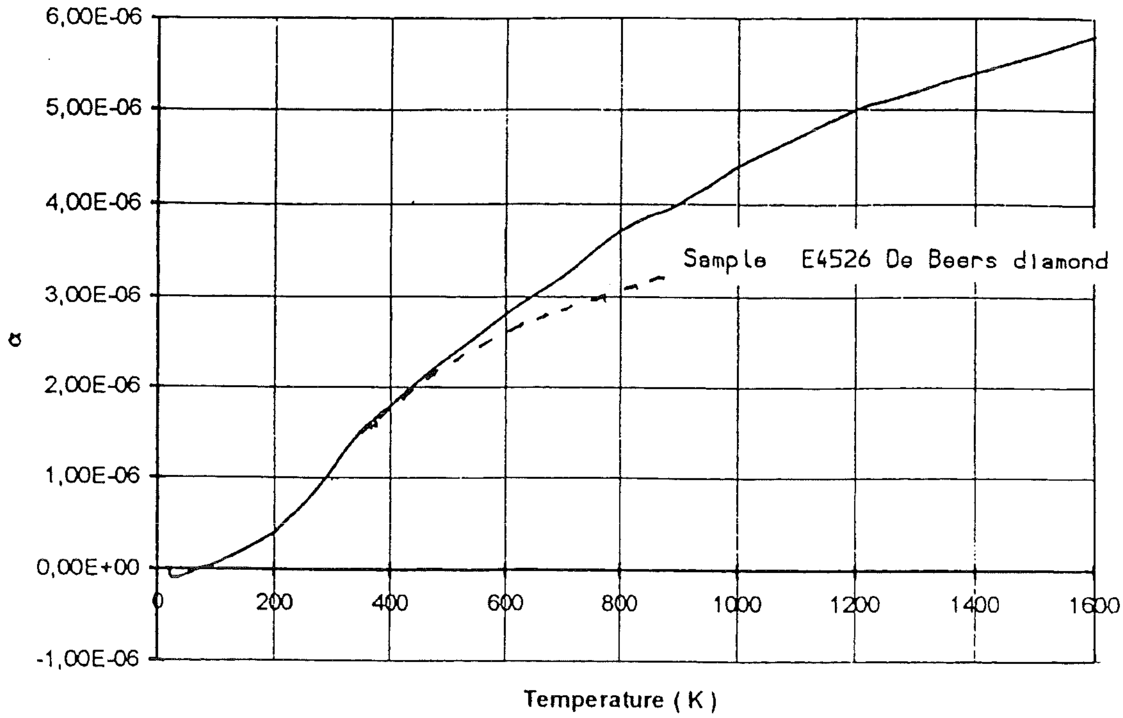


Fig. 2: Temperature dependence of thermal expansion coefficient α of diamond.

Only Kovar, an Iron-Nickel-Cobalt alloy, Molybdenum and Tantalum are ferrule materials with a thermal expansion nearly as low as that of CVD diamond but these materials are difficult to machine.
The metallization and brazing tests described in the following paragraphs have been performed in collaboration of EU Industry DeBeers (Charters, UK) and FZK.
Based on these experiences we decided to use the following strategy to develop adequate bonding and brazing techniques.

Step 1 (GB6-EU-T245/6)
Metallization/Bonding Techniques for 450°C Bakeout:

Usage of Inconel 600 as ferrule material which is even easier to machine compared to the usual Inconel 625 and very easy to weld to stainless steel. Electron-beam or laser-beam welding is also no problem. The elastic properties of a ductile Al-based braze joined by solid-phase diffusion bonding to the CVD-diamond disk are used to reduce the thermal stressing as much as possible. The disadvantage of this technique is the relatively low allowed bakeout temperature of the window unit of 450°C (guaranteed by DeBeers) and the brazing joint is prone to corrosion when exposed to cooling water. On the other hand, in a closed circuit cooling system the corrosion resistance can be improved by using the CC.15 Cooling Water Treatment (Sodium Nitride, Sodium Metasilicate, Sodium Borate and Sodium Mercapto-benzothiazole) as corrosion inhibitor. This treatment should be used with deionized or demineralized water to be fully effective. Dosage is calculated at 1 liter of CC.15 to 130 liters of water. The treatment should be checked every three months using a testing kit which the suppliers can provide. Costs are currently in the region of 100 US $ per 25 liter container.

Since grey (cheap) diamond shows the same thermal expansion and thermal shock behavior like white (expensive) diamond, for the first bonding and brazing experiments grey diamond disks, with step by step increasing diameter and thickness, were brazed to a circular Inconel 600 sleeve welded to a CONFLAT (CF) flange for vacuum tests. The dimensions of the CVD-diamond disks were:

25 mm diameter/ 0.5 mm thickness,
48 mm diameter/ 0.5 mm thickness and
100 mm diamater/ 1 mm thickness.

The test windows were thermally cycled in a UHV vacuum oven (pressure at 470°C: $5 \cdot 10^{-7}$ mbar) as if they would be installed as a window on a gyrotron (see Fig. 3). According to the supplier, the assembly should be uniformly heated to a maximum of 450°C at a rate of maximal 2°C per minute (we had 470°C and 0.5°C per minute) to reduce any thermal stressing across to whole mounting as much as possible.

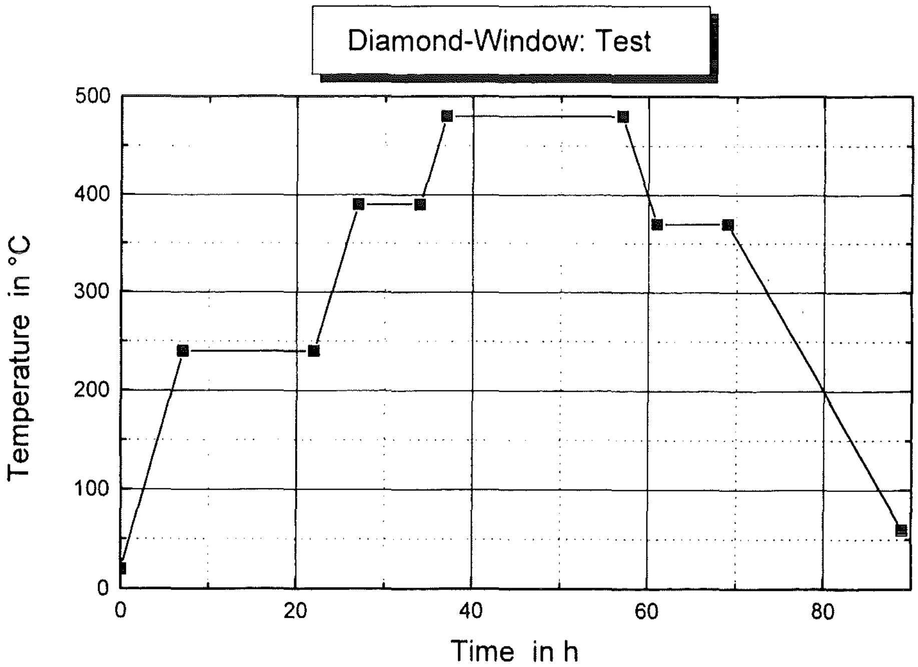


Fig. 3: Thermocycle applied to test diamond windows in a UHV vacuum oven.

The temperature cycling tests with the different test windows mounted to CF flanges or Inconel 600 waveguide cylinders (70 mm inner diameter) brazed to both sides of a grey-diamond disk with 100 mm outer diameter and 0.8 mm thickness to from part of a full window assembly, were very successful. The Inconel cylinders were strengthened by Molybdenum rings in order to reduce thermal expansion during bakeout. The leackage rate before and after bakeout was determined to be approximately 10^{-9} mbar $\cdot$ l/s which is the measuring limit of our He-leak tester. No diffusion degrading of the brazing has been observed.
Following these experiments using grey-diamond test disks, two white-diamond window disks where mounted and tested in the same manner:

EU: outer diameter 100 mm, aperture diameter 80 mm, thickness 1.60 mm (resonance at 118 GHz)

JA: outer diameter 96 mm, aperture diameter 83 mm, thickness 2.23 mm (resonance at 170 GHz)

The experiments at JAERI showed that baking at 450°C for 2 days (flat top) is o.k., but leakage of the brazing occured for longer flat top times.

Step 2 (GB6-EU-T360)
Metallization/Bonding Techniques for 550°C Bakeout:

In order to shorten the conditioning time of the gyrotron after manufacture, microwave industries prefer a bakeout temperatur of 550 °C which is too high for the Al-based brazing. Additionally, the braze should be less prone to corrosion when exposed to the cooling water.
First tests have been made on metallized white-diamond disks which have been bounded with Au-based brazing on both sides to Inconel 600 cylinders to form part of a full window assembly (outer disk diameter = 30 mm, disk thickness 0.8 mm, window aperture = 24 mm). The Inconel cylinders are strengthened by Molybdenum rings in order to reduce thermal expansion during bakeout. These sub-assemblies have been subjected to the bakeout cycle of a gyrotron up to 550 °C without any degradation. In a next step the outer CVD-diamond disk diameter was increased to 50 mm, the window aperture to 40 mm and the disk thickness to 1.8 mm (Fig. 4). These sub-assemblies have also been subjected to the bakeout cycle of a gyrotron up to 550°C with the result that the bonding at both sides got a leak. This means that the Au-based braze is not as elastic as the former Al-based braze that is only bakeable up to 450°C. The next bonding/brazing tests will be performed either with Molybdenum waveguide sleeves (small thermal expansion) or with Copper waveguide sleeves (elastic) using appropriate brazing material.

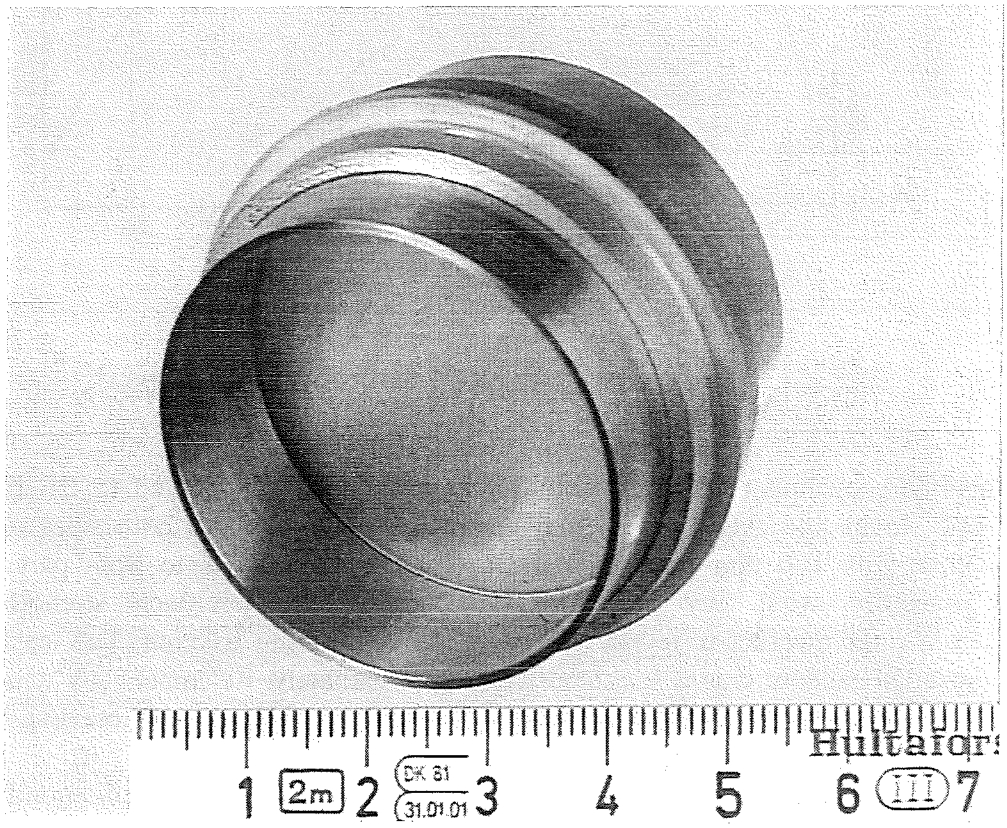


Fig. 4: Photograph of CVD-diamond bakeout test window unit.

2. Fabrication of a Single-Disk CVD-Diamond Window for 118 GHz, 0.5 MW, 210 s (Subtask 2.2)

The design of a 118 GHz, 0.5 MW, 210 s CVD-diamond window was finished and the window unit has been manufactured by Thomson Tubes Electroniques (TTE). Fig. 5 shows the layout of the window unit.

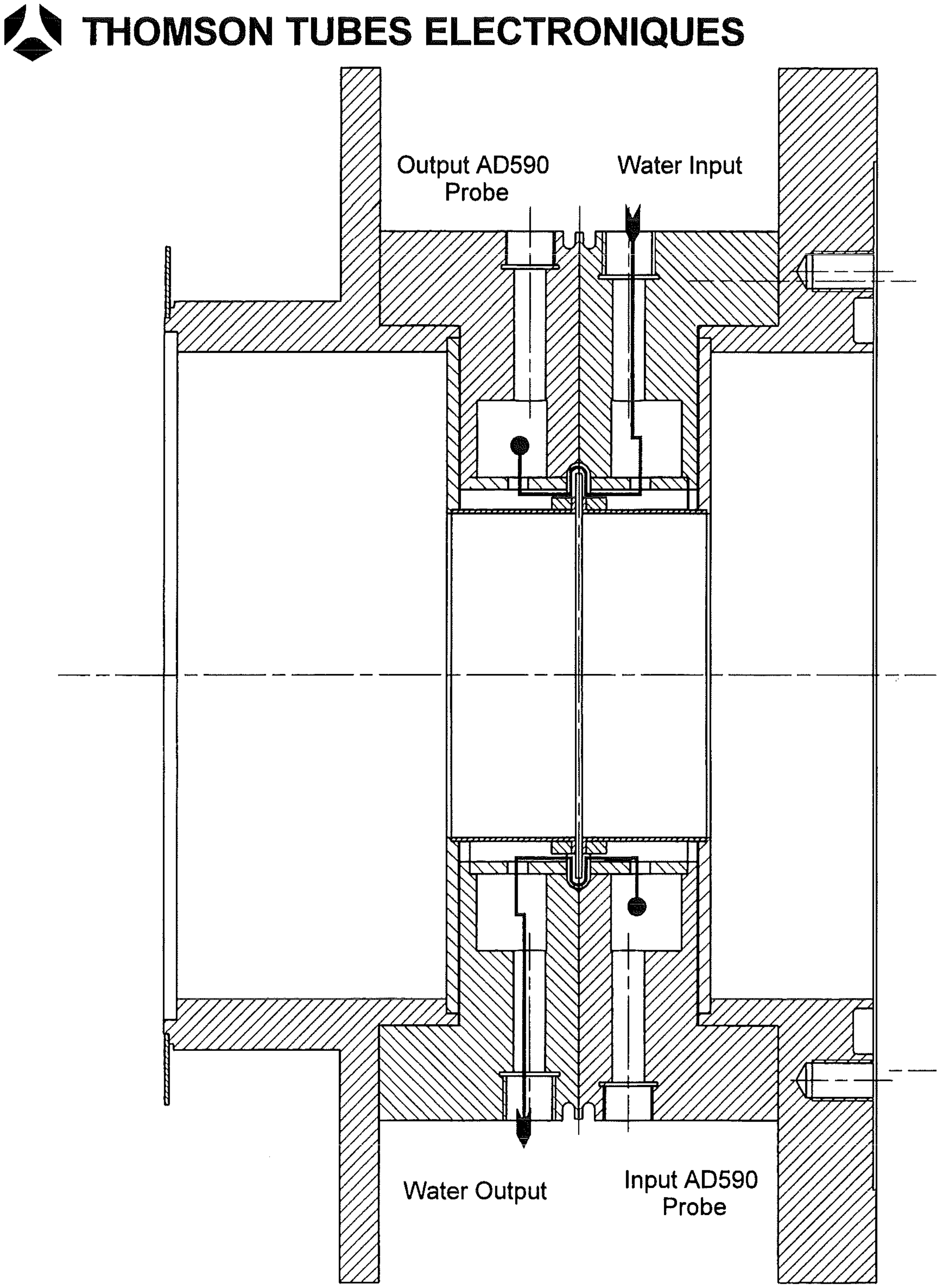


Fig. 5: Layout of the 118 GHz, 0.5 MW, 210 s CVD-diamond window.

The universal design allows tests in an evacuated HE_{11}-transmission line (inner diameter I.D.= 63.5 mm) at CEA Cadarache and also direct mounting to a 118 GHz TTE gyrotron. The window has an aperture of 80 mm (100 mm outer disk diameter), a thickness of 1.6 mm, a loss tangent of only $6 \cdot 10^{-6}$ (world record!), a thermal conductivity of 2050 W/mK (at room temperature, but decreasing for increasing temperature) and has also been bonded using the Al-braze technique. Fig. 6 and 7 show a drawing of the cross section and a photograph of the CVD-diamond disk bonded to the two Inconel 600 cuffs and strengthened by two Molybdenum rings in order to reduce thermal expansion during bakeout procedures. Fig. 8 shows the dependence of the reflectivity of the 1.6 mm diamond disk on frequency and in Fig. 9 the distribution of the loss tangent (tanδ) measured at 145 GHz across the disk are plotted.

The Figures demonstrate a 1 % power reflection bandwidth of 2.6 GHz and a sufficient homogeneity of tanδ. Finite element calculations including brazing/bonding stress show that the maximum principal stress is located in the window brazing and is always present. Because the ultimate bending strength of a white CVD-diamond disk with 1.6 mm thickness is approximately 600 MPa (see 6.4) all stresses are well below the admissible limit. Fig. 10 shows the time dependence of the center and edge temperatures for a cooling water temperature of 20°C and a heat transfer coefficient of 12 kW/m²K. The simulations reveal that steady state conditions are generally achieved in less than 3 s (central temperature: 28.5 °C; edge temperature: 21.3°C). The window will be tested in September 1998 at CEA Cadarache when the long-pulse 118 GHz gyrotron will be available. The experimental arrangement for these measurement is plotted in Fig. 11.

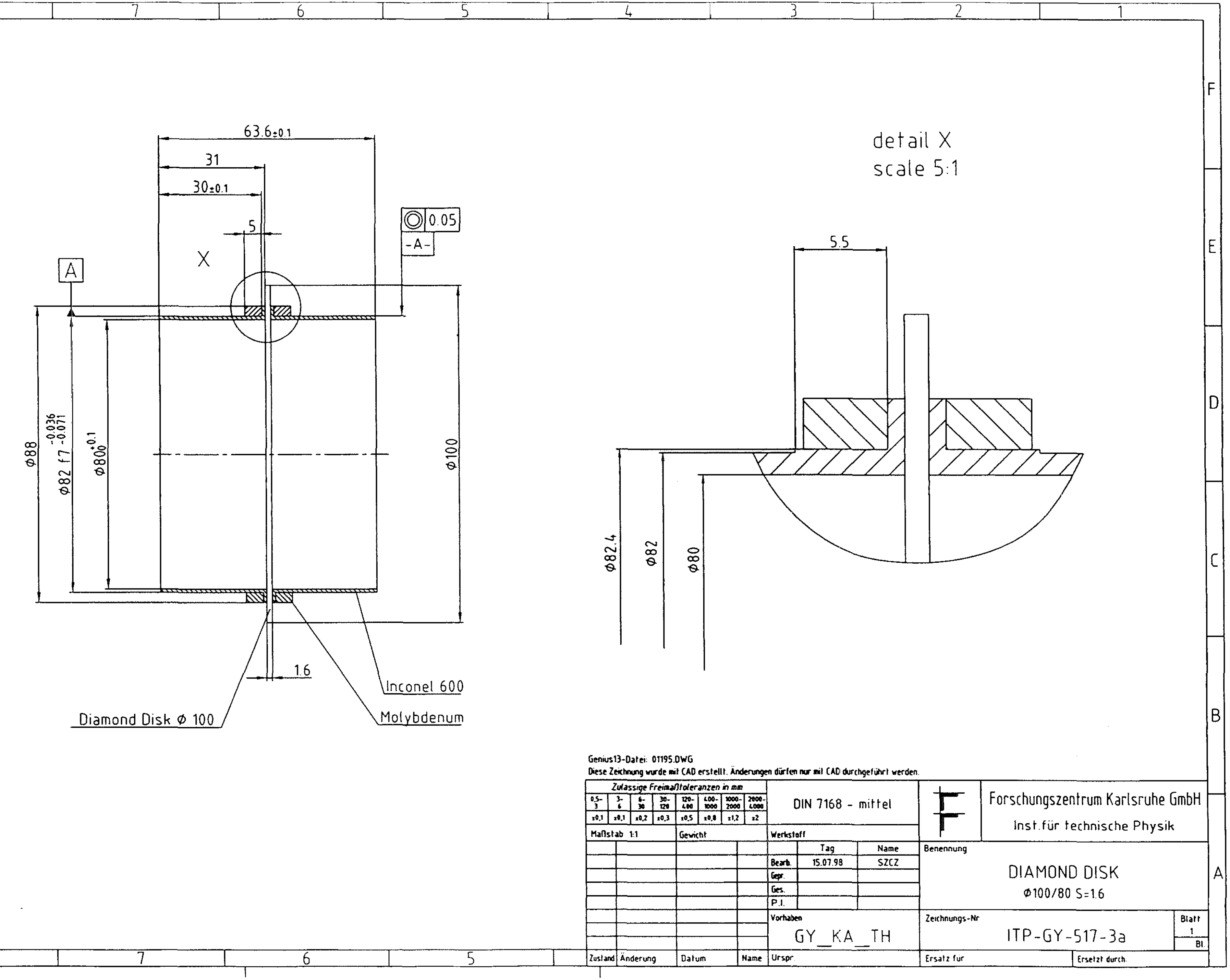


Fig. 6: Cross-section of the bonded CVD-diamond disk.

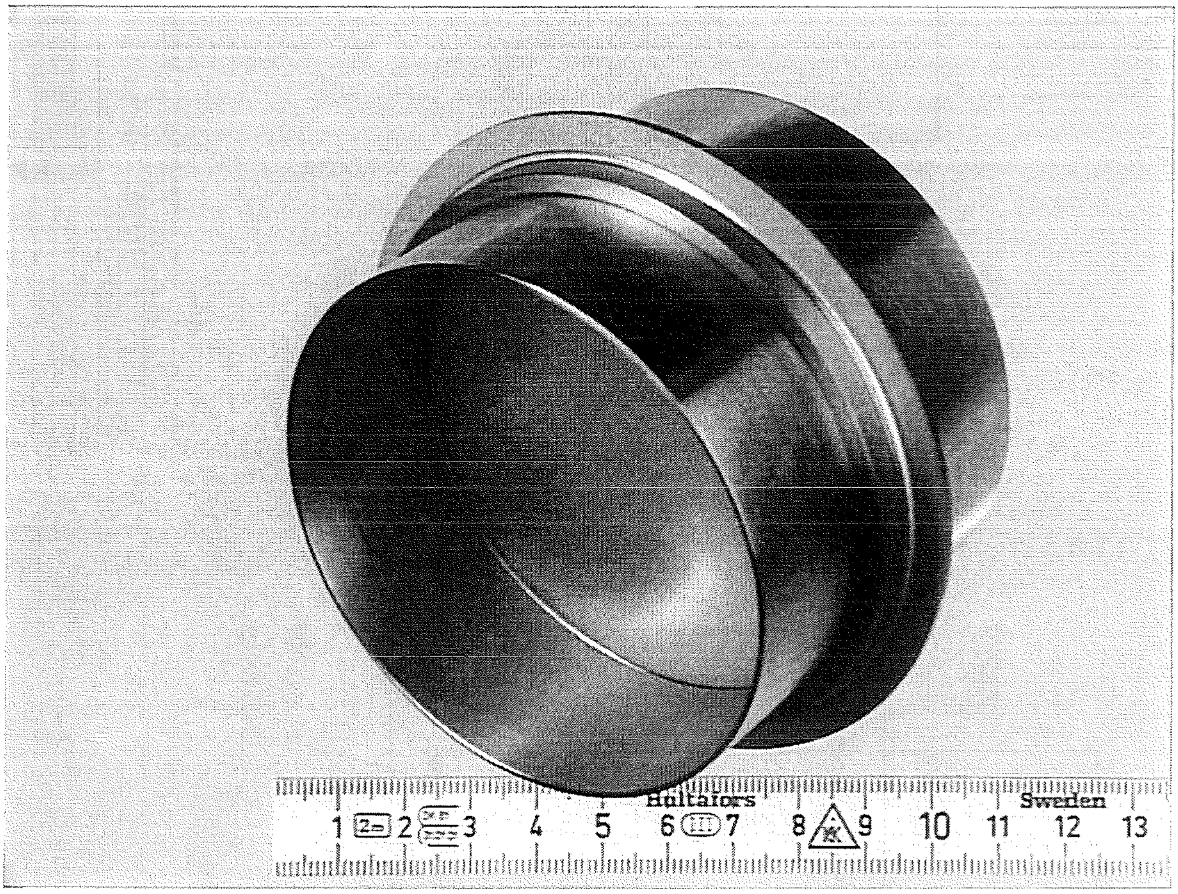
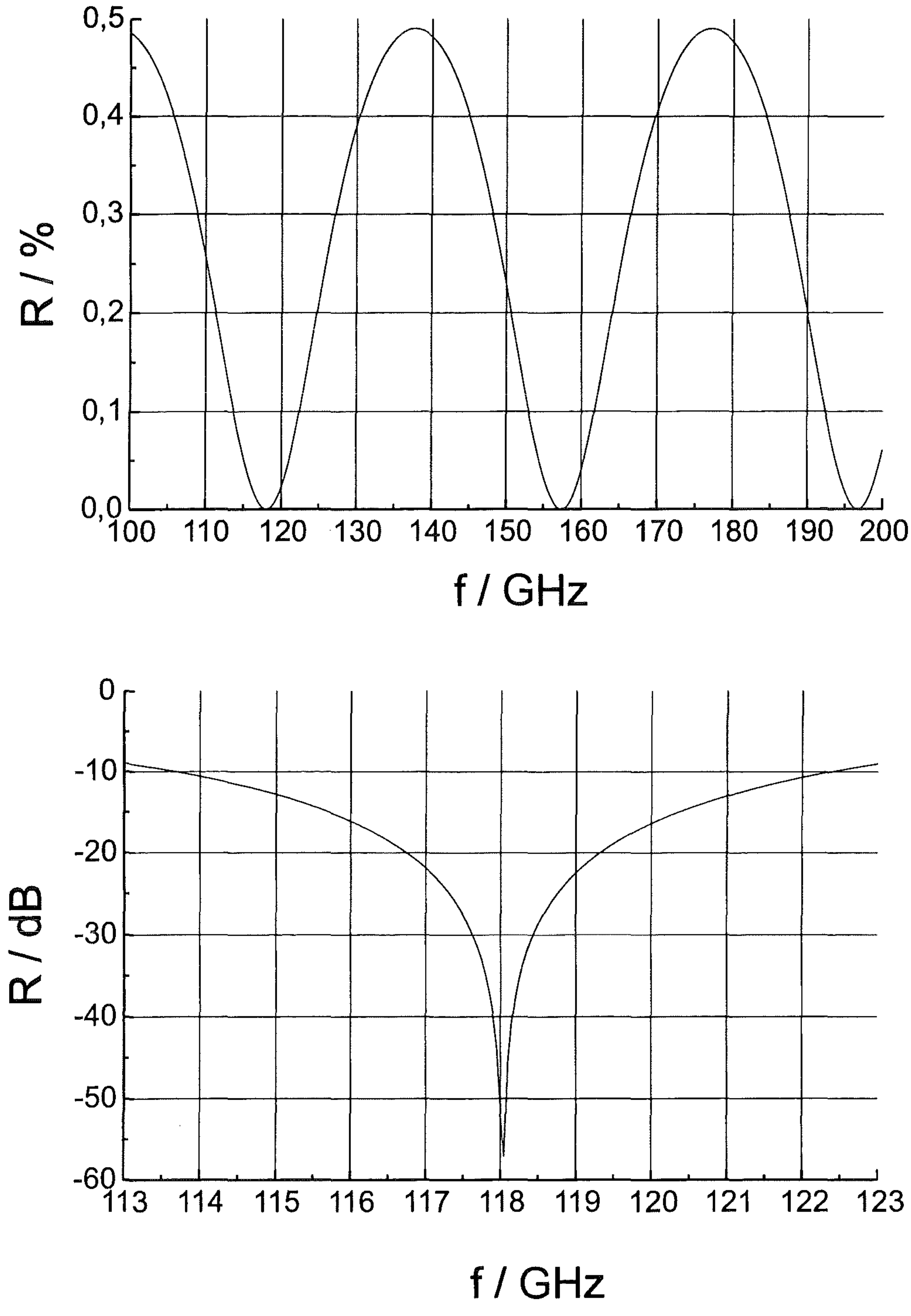


Fig. 7: Photograph of the CVD-diamond disk "Super-FZK" mounted to two Inconel 600 sleeves.

Reflectivity of 1.6 mm diamond window



d = 1.6 mm; ε_r = 5.67; tanδ = $6 \cdot 10^{-6}$

Fig. 8: Frequency dependence of reflectivity of the 1.6 mm thick CVD diamond disk.

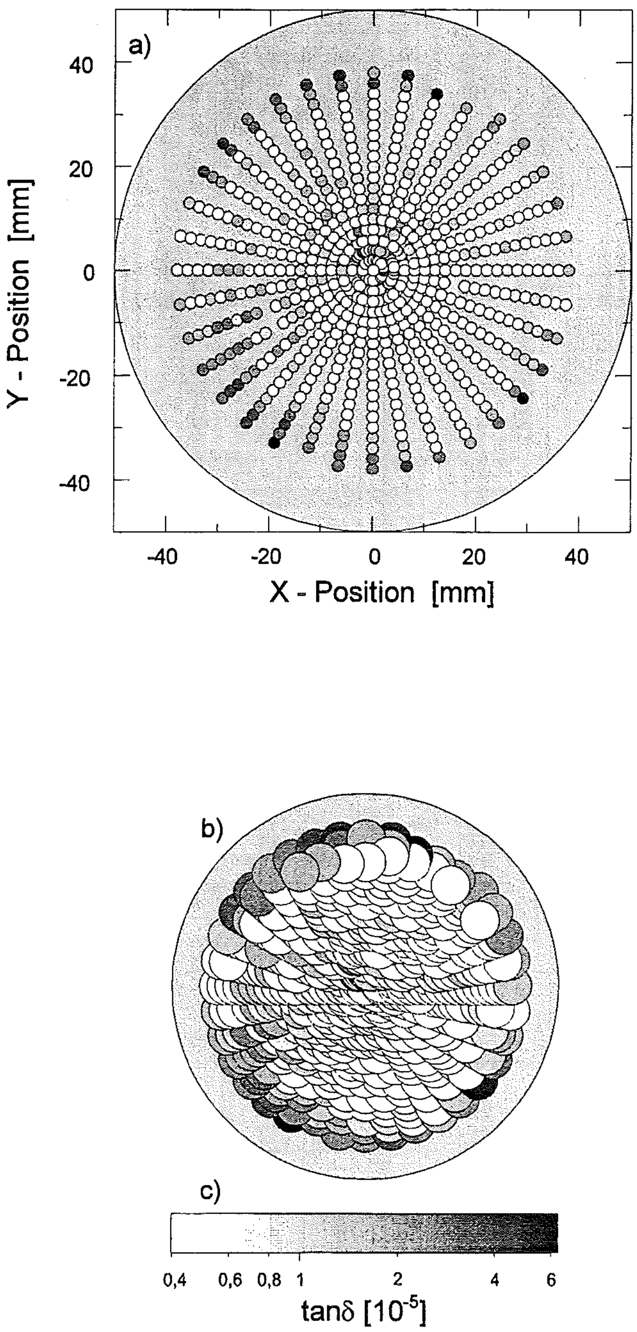


Fig. 9: Distribution of loss tangent values of the CVD-diamond window disk "Super-FZK".

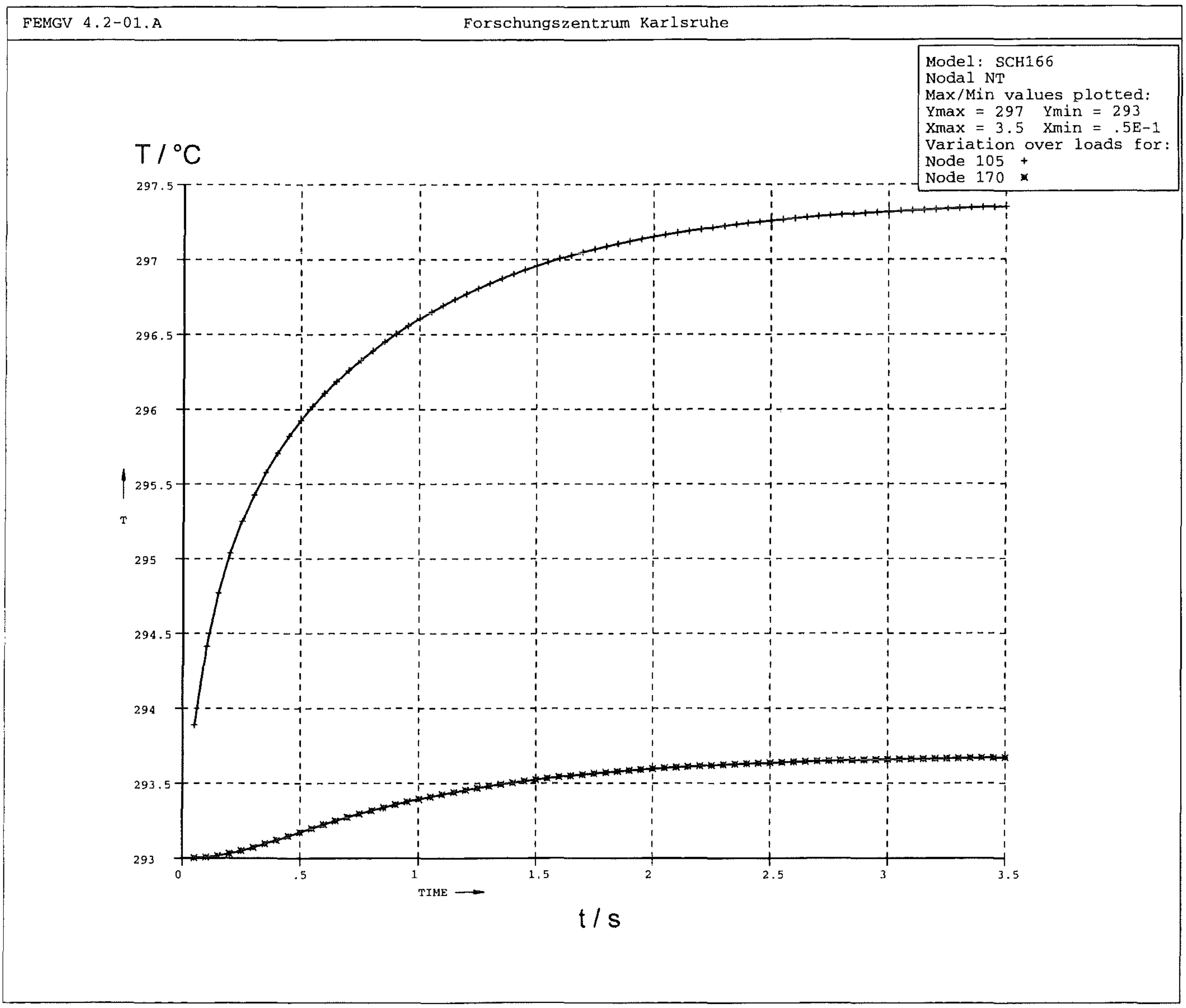
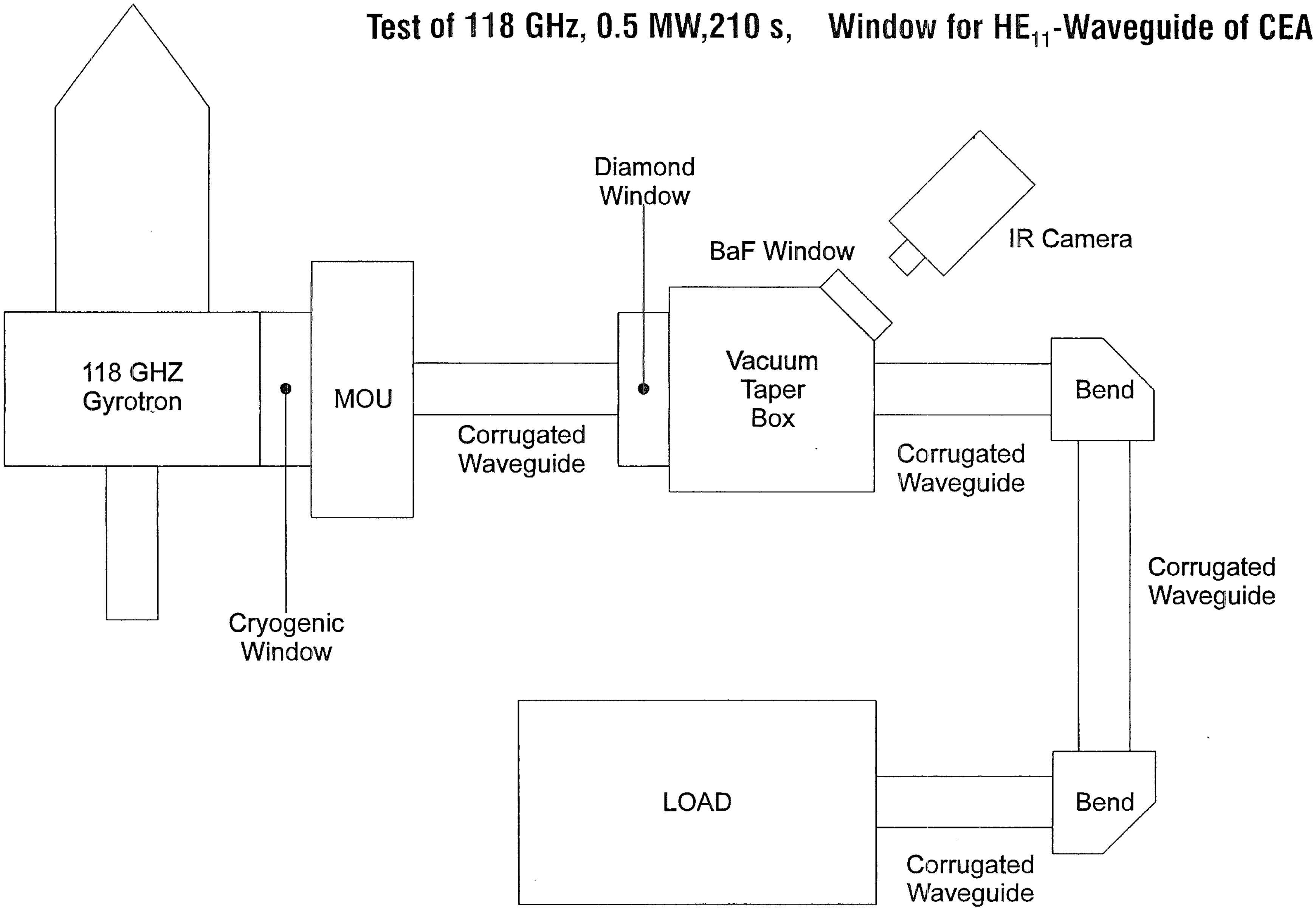


Fig. 10: Time dependence of center and edge temperatures of CVD-diamond window at 0.5 MW, 118 GHz (cooling water at 293 K).



THOMSON TUBES ELECTRONIQUES

Fig. 11: Experimental arrangement for the test of the 118 GHz, 0.5 MW, 210 s CVD-diamond window in an evacuated HE_{11}-waveguide at CEA Cadarache.

3. High-Power CVD-Diamond Window Test (Subtask 2.3)

High-power tests on a 170 GHz gyrotron equipped with a CVD-diamond window were performed in collaboration with the JA Home Team at JAERI. In order to check the usability of such a polycrystalline diamond disk with a diameter of 96 mm and a thickness of 2.23 mm, the material properties determined by means of resonator measurements have been confirmed in a first series of high power experiments. For this reason the unbrazed disk has been placed in the millimeter wave beam of the gyrotron. The edge temperature increase after transmission of a 1 s rf pulse with a power level of 110 kW was measured and compared with numerical simulations. The values of loss tangent ($1.3 \cdot 10^{-4}$) and thermal conductivity (1800 W/mK at room temperature) were confirmed (GB6-EU-T245/6).

After successful metallization of the edge it was possible to braze the disk as an ultra high vacuum barrier into a stainless steel window housing. For cooling purposes the window assembly is equipped with a cooling channel which enables water at room temperature to flow around a small edge segment of the disk. The remaining window clear aperture is 83 mm. To prove the efficiency of this cooling system the complete window assembly again was placed in the Gaussian like rf output beam of the gyrotron. By transmitting an rf power of 110 kW for a pulse duration of 10 s the steady state condition of the center temperature at a level of 60 °C was reached after 7 s by applying a water flow rate of 11 l/min (see Annex 1).

After this successful pre-test the window assembly was finally mounted directly as the rf output window on the gyrotron oscillator itself. During the following experiments for the very first time the operation of this high-power millimeter-wave source has not been limited by the power transmission capability of the required vacuum window.

By transmitting an rf power of 0.45 MW for 8 s (3.6 MJ) the maximum window center and edge temperatures were measured to be 150 °C and 13 °C, respectively, with a saturation time of approximately 5 s [1]. The temperature of the cooling water was 10°C.

As an example for such a continuous wave like operation Fig. 12 shows the dependence of the window center temperature versus time. For this measurement the maximum available output power of 0.52 MW was used. The gyrotron operation in this case had to be stopped after 6.2 s due to the fact that the critical gas pressure inside the tube had been reached.

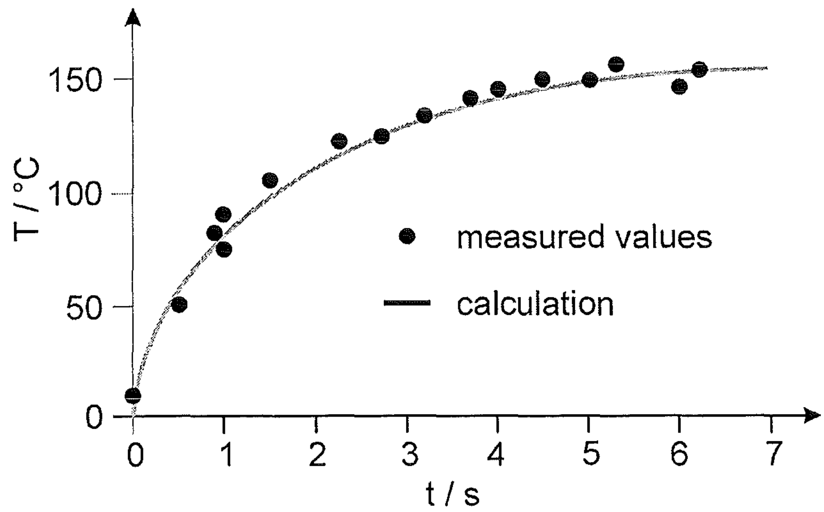
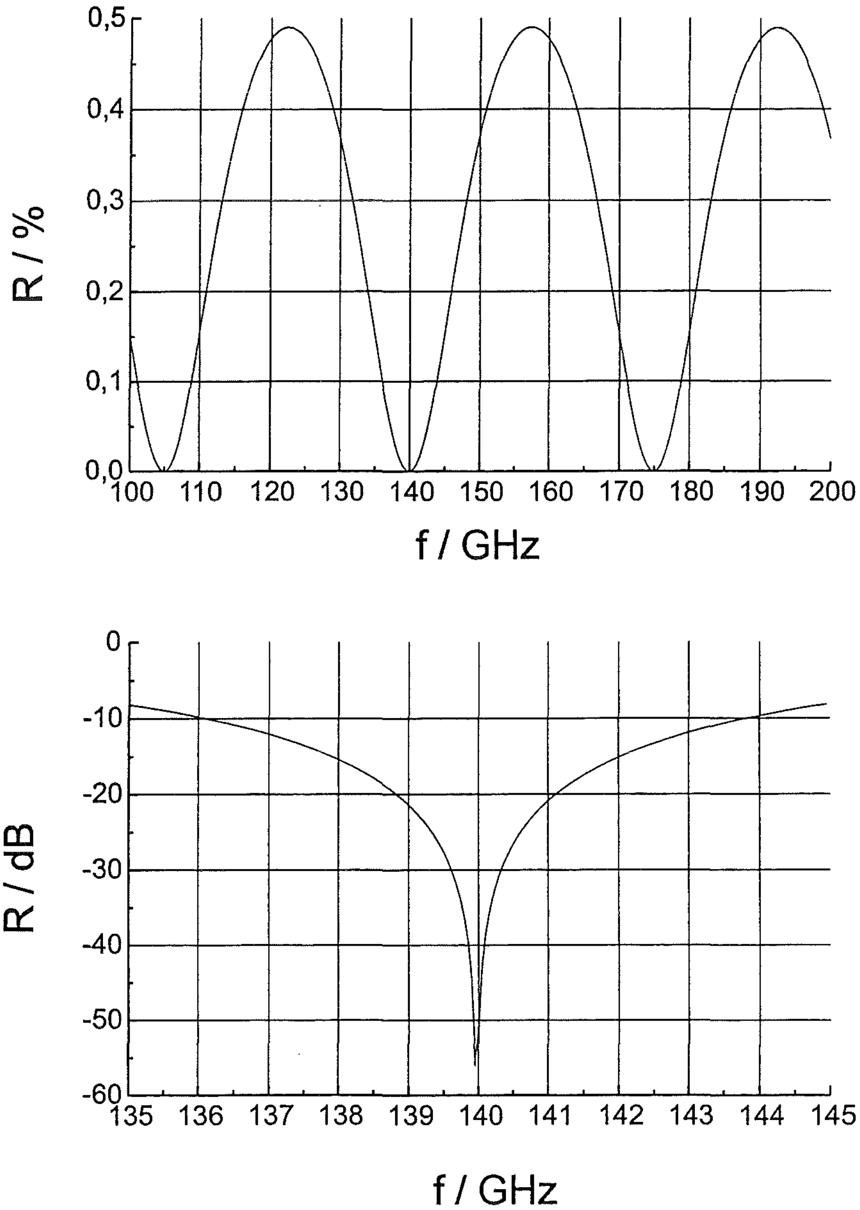


Fig. 12: Window center temperature increase versus time during transmission of a flattened rf beam at a frequency of 170 GHz and power level of 0.52 MW [1].

4. Preparation of a Single-Disk CVD-Diamond Window for 140 GHz, 1 MW, CW (Subtask 2.4)

Three CVD-diamond disks with 106 mm outer diameter and 1.8 mm thickness have been ordered at DeBeers for the development of a 140 GHz, 1 MW, CW prototype gyrotron for W7-X with a single-stage depressed collector (efficiency > 45 %). The gyrotron window will have an aperture of around 86 mm.
Fig. 13 reveals a 1 % power reflection bandwidth of 2.3 GHz.



$d = 1.799$ mm; $\varepsilon_r = 5.67$; $\tan\delta = 2 \cdot 10^{-5}$

Fig. 13: Frequency dependence of the reflectivity of a 1.8 mm thick CVD-diamond disk.

Fig. 14 shows the calculated maximal (center) and minimal (cooling edge) temperatures of the diamond disk as a function of the width of the cooling rim at a mm-wave power of 1 MW, 140 GHz in the fundamental Gaussian mode (beam radius w = 26 mm). A cooling rim of about 3 mm width is sufficient to get a maximum center temperature of only around 63 °C and an edge temperature of not more than 32°C. A loss tangent of $2\cdot10^{-5}$, a thermal conductivity of 1800 W/mK (at room temperature) and a heat transfer coefficient of 12 kW/m²K to the cooling water (T = 20°C) have been taken into account. These FE simulations also show that the steady state conditions are generally achieved in approximately 4 s (Fig.15).

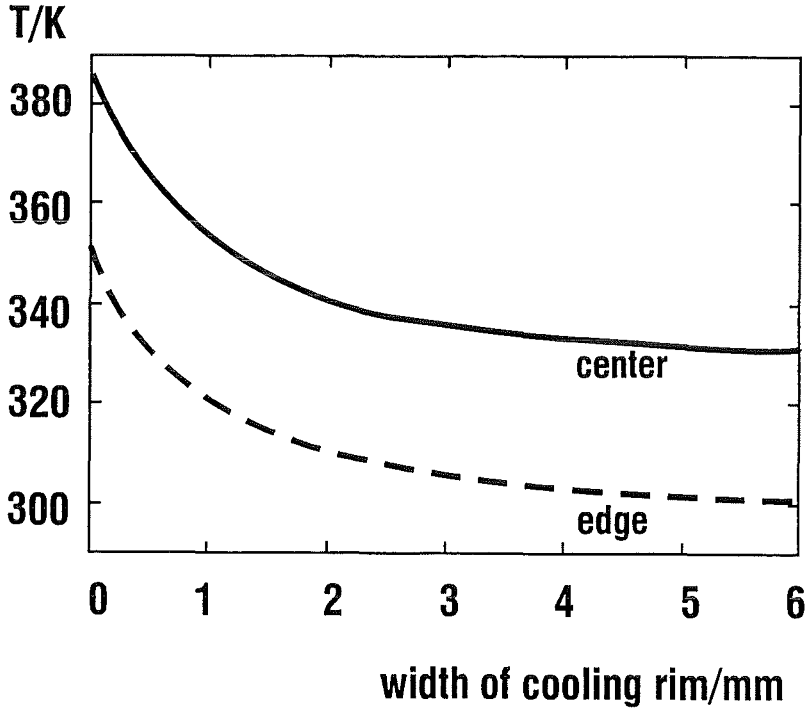


Fig.14: Dependence of center and edge temperatures of CVD-diamond window on the width of the applied cooling rim at 1 MW, 140 GHz.

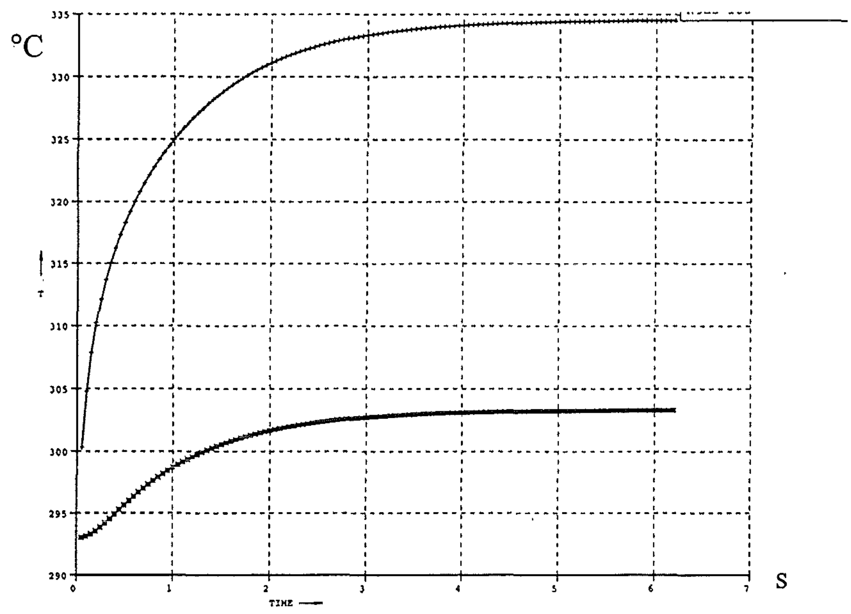


Fig. 15: Time dependence of center and edge temperatures of CVD diamond window at 1 MW, 140 GHz (width of cooling rim = 4 mm).

5. Neutron Irradiation Effects on CVD Diamond (Subtask 2.5)

The effect of radiation damage on the material properties of ceramics is a general issue for components used in heating and current drive and in diagnostic systems. The main efforts in this field are performed within the ITER task T246 which have been documented recently for the period of 1995 – 1998 in a special final report [2].

For the present ITER task T360 on ECH window development, the research activities are focused to investigate special grades of CVD diamond which fulfil particularly the requirements for ECH window uses, which include also availability of large sized disks, and their potential tolerance limits with respect to radiation damage.

In the previously reported tasks [3] on ITER window development (GB6-EU-T245/6), the first data of dielectric loss at 145 GHz were given for CVD-diamond grades subjected to electron and neutron irradiations which produced structural damage of 10^{-5} dpa. The absence of significant radiation-increased losses allowed to define a radiation programme which will lead in the end to so-called "technological irradiations" where actual window disks will be irradiated at an established maximum fluence limit.

The steps taken follow the previous experience gained for establishing a recommended fluence limit for cryogenically cooled ECH windows based on sapphire. This programme was finalized by two neutron irradiations at cryogenic temperatures (Table 1), which define the tolerable level to $0.4 - 1.0 \times 10^{21}$ n/m^2 in that case.

The series of neutron irradiations setting the target fluence for the technological irradiation are performed with smaller specimens. The specimens are placed in Cd-screened irradiation capsules which are flooded by the coolant in the reactor pool at GKSS (Geesthacht, D). Apart from specimens that have disk-shaped geometries adapted to dielectric measurements (30 or 40 mm diameter disks), these irradiation tasks, named "Ambient I – III", are scheduled to take also pellet-shaped specimens (12 mm diameter disks) for thermal conductivity measurements. However, because of pronounced discrepancies between results from thermal conductivity measurements performed with the laser-flash and the photo-acoustic method after irradiation "Ambient I" – which investigated either pellets or disks – the irradiation "Ambient II" was split into two projects. Whereas the first one contained only disks, the second one was loaded with corresponding pellets. As the latter specimens have just been received back to the laboratories, the discussion of radiation effects on thermal conductivity has to wait for the concluding tests and is not part of this report.

The irradiation "Ambient I" contained disks bought from DeBeers which covered the material development until the demonstration of the scale-up potential of CVD diamond (Table 2). The specimen representing the latter step (DB6) was selected together with a more recent specimen (DB7) cut out of an actual 4 inch window disk for irradiation "Ambient II". Already the visual inspection of these specimens gave evidence of a pronounced irradiation effect: the original fully clear appearance of DB7 and the greenish colour of DB6 (after receiving 10^{-5} dpa) both shifted to an intense black coloration at the damage level of 10^{-4} dpa.

But still for dielectric loss measurements at 145 GHz and room temperature, no dramatic degradation in the loss tangent is observed. With a new installation for dielectric measurements at 90 GHz, further evidence for the minor degree of radiation-induced changes can be given (Fig. 16). There seems to be a general trend that inhomogeneities, like differences in the contributions from the growth and nucleation sides tend to disappear at increasing damage levels. However, these changes, and also possible frequency dependences, are difficult to extract from the uncertainty bounds of the measurements. Temperature variable measurements show no significant differences in the curves of the two specimens before and after irradiation (Fig. 16), which also holds for the permittivity (Fig. 17). The loss data for specimen DB7 are in a way approximative as the extremely low loss levels observed in the dedicated room temperature measurements could not be well reproduced at the related temperature points of the temperature variable measurements. The reason for this discrepancy is still open. Yet, the absence of any severe and detrimentral effect of the neutron irradiation on dielectric absorption at a fluence of 10^{21} n/m^2 is without any doubt. As this level was the recommended fluence limit for sapphire, it seems to be the most appropriate one for the first technological irradiation under preparation. Further insight on radiation resistance is to be expected from the forthcoming irradiation Ambient III which takes again DB6 and DB7 as relevant specimens.

Table 1: Scope of the FZK neutron irradiation studies within the ITER/NET tasks on 'Ceramics for Heating and Current Drive Systems' (1995 - 1998).

Name of irradiation	Dielectric specimens	Thermophys. specimens	T_{irr}	Fluence (E > 0.1 MeV)	Irradiation time	Status
Cryo I (Petten NL)	HEMEX U. C. Sapphire Quartz	as dielectric specimens	77 K	10^{21} n/m^2	2 - 3 days	Irradiation performed: July 1995 Results published
Cryo II (Garching, D)	no specimens insufficient capsule size	U. C. Sapphire	4.2 K	10^{20}n/m^2	< 2 - 3 weeks	Irradiation performed: December 1996 Results reported to ITER
Ambient I (Geesthacht, D)	CVD Diamond Doped Silicon Pure Silicon	CVD Diamond Doped Silicon Pure Silicon	~330 K	10^{20} n/m^2	2 - 3 days	Irradiation performed: November 1996 Results published
Ambient II (Geesthacht, D)	as in Ambient I	as dielectric specimens	~330 K	10^{21} n/m^2	2 - 3 days	Irradiation performed: March 1998 Studies performed
Ambient II a (Geesthacht, D)	no specimens	CVD Diamond Pure Silicon	~330 K	10^{21} n/m^2	2 - 3 days	Irradiation performed: July 1998 Specimens received
Ambient III (Geesthacht, D)	as in Ambient I	as dielectric specimens	~330 K	10^{22} n/m^2	≈ 3 weeks	Irradiation under preparation targeted for Sept. 98
Technological I (Petten, NL)	CVD Diamond (window size)	as dielectric specimens	~300 K	10^{21} n/m^2	≈ 1 week	Irradiation foreseen in 1999

Table 2: Pre- and post irradiation studies on specially developed CVD-diamond grades for high power EC windows.

a) Frequency: 145 GHz

Internal specimen code	Pre-irradiation studies dielectric loss tanδ [10^{-4}]	Post-irradiation studies at ≈10^{-5} dpa (Amb I)		Post-irradiation studies at ≈10^{-4} dpa (Amb II)	
		"growth face in " tanδ [10^{-4}]	"nucl. face in" tanδ [10^{-4}]	"growth face in" tanδ [10^{-4}]	"nucl. face in" tanδ [10^{-4}]
DB 1 (evaluation set)	1.2 (± 0.5)	0.85 (± 0.15)*	0.85 (± 0.15)*	-	-
DB 5 (refinement set)	0.9 (± 0.5)	0.60 (± 0.15)	0.65 (± 0.15)	-	-
DB 6 (scale-up grade)	0.2 (± 0.1)	0.20 (± 0.05)	0.45 (± 0.05)	0.30 (± 0.05)	0.30 (± 0.05)
DB 7 (window grade)	0.10 (± 0.05)	-	-	0.20 (± 0.10)	0.20 (± 0.10)

* Electron irradiation (E. Hodgson/CIEMAT)

b) Frequency: 90 GHz

Internal specimen code	Pre-irradiation studies dielectric loss tanδ [10^{-4}]	Post-irradiation studies at ≈10^{-5} dpa (Amb I)		Post-irradiation studies at ≈10^{-4} dpa (Amb II)	
		"growth face in " tanδ [10^{-4}]	"nucl. face in" tanδ [10^{-4}]	"growth face in" tanδ [10^{-4}]	"nucl. face in" tanδ [10^{-4}]
DB 6 (scale-up grade)	-	0.35 (± 0.10)	0.45 (± 0.10)	0.30 (± 0.05)	0.30 (± 0.05)
DB 7 (window grade)	0.10 (± 0.05)	-	-	0.20 (± 0.05)	0.25 (± 0.05)

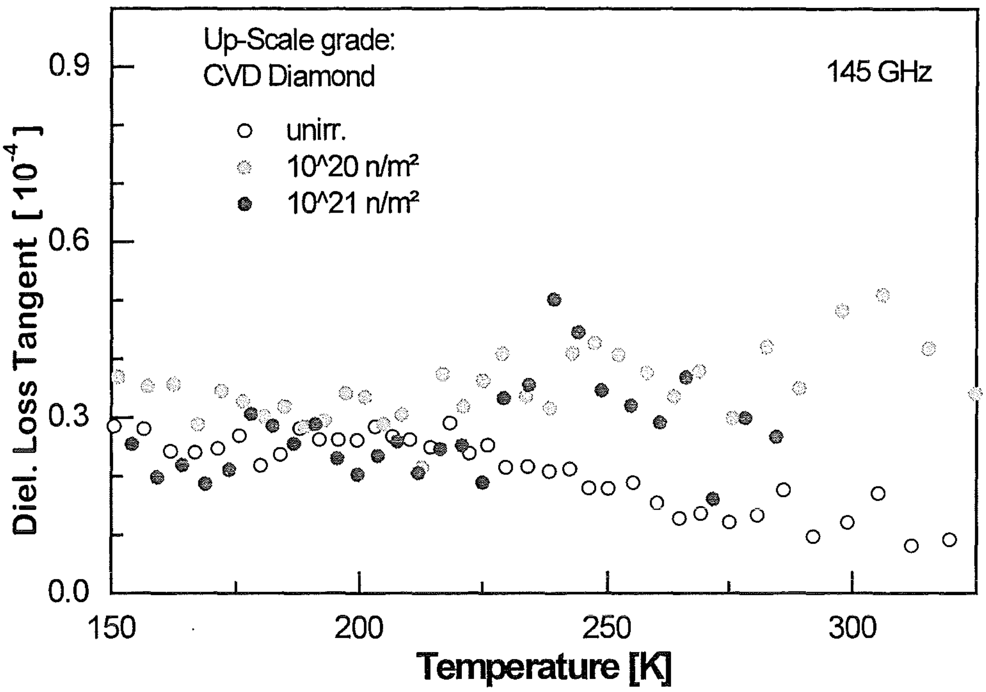


Fig.16a: The temperature dependence of dielectric losses observed at 145 GHz in the scale-up grade CVD diamond material before and after irradiation with fast neutrons.

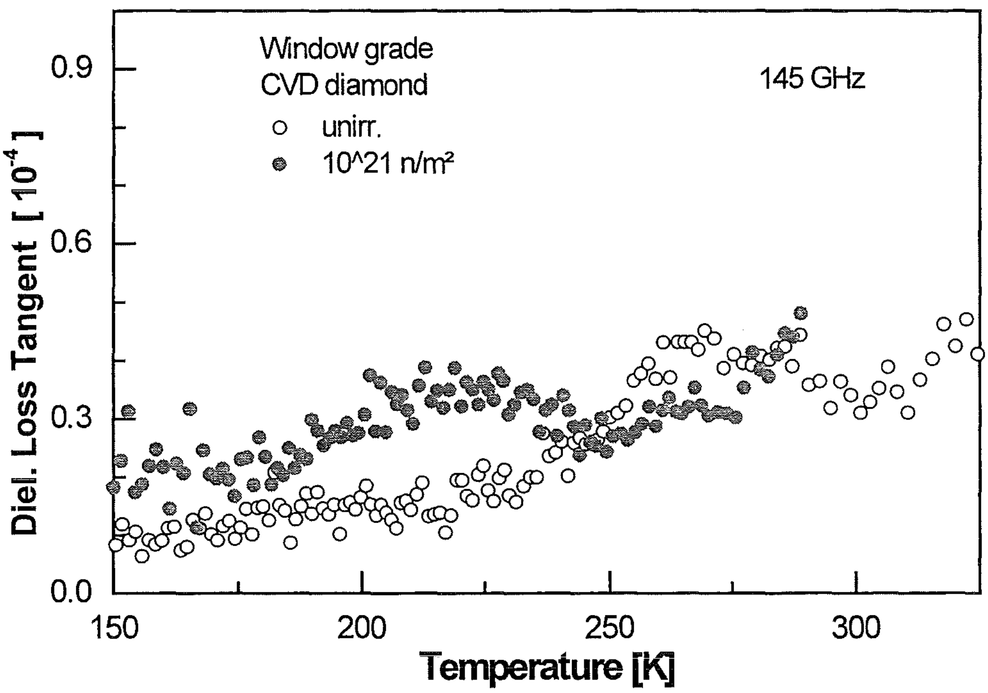


Fig.16b: The temperature dependence of dielectric losses observed at 145 GHz in the window grade CVD diamond material before and after irradiation with fast neutrons.

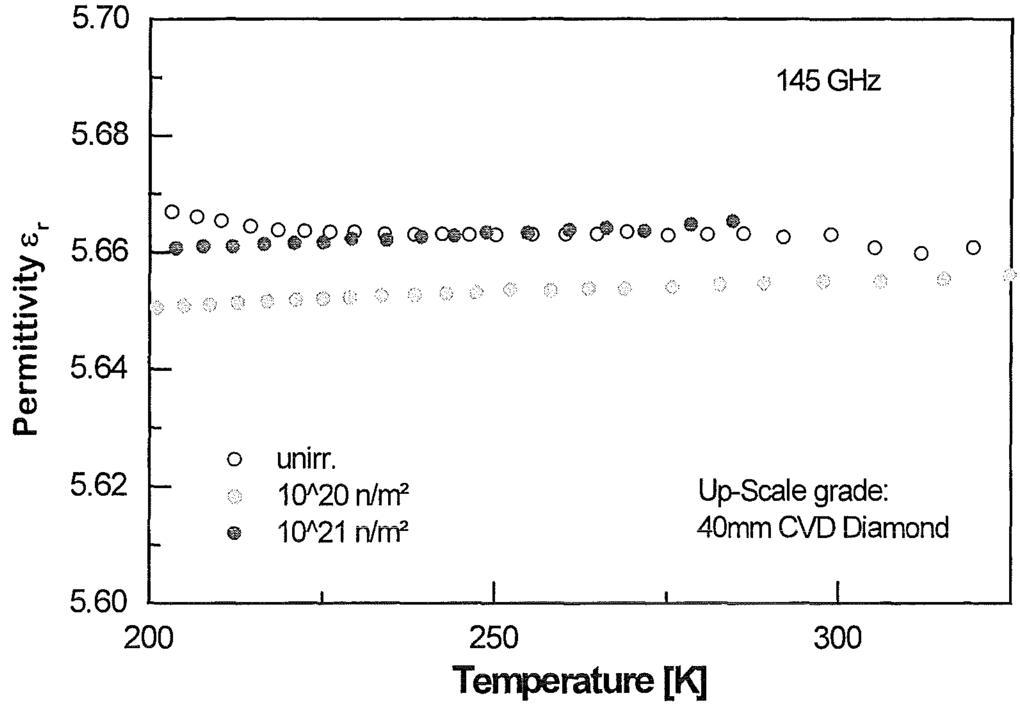


Fig. 17a: The temperature dependence of the permittivity observed at 145 GHz in the scale-up grade CVD diamond material before and after irradiation with fast neutrons.

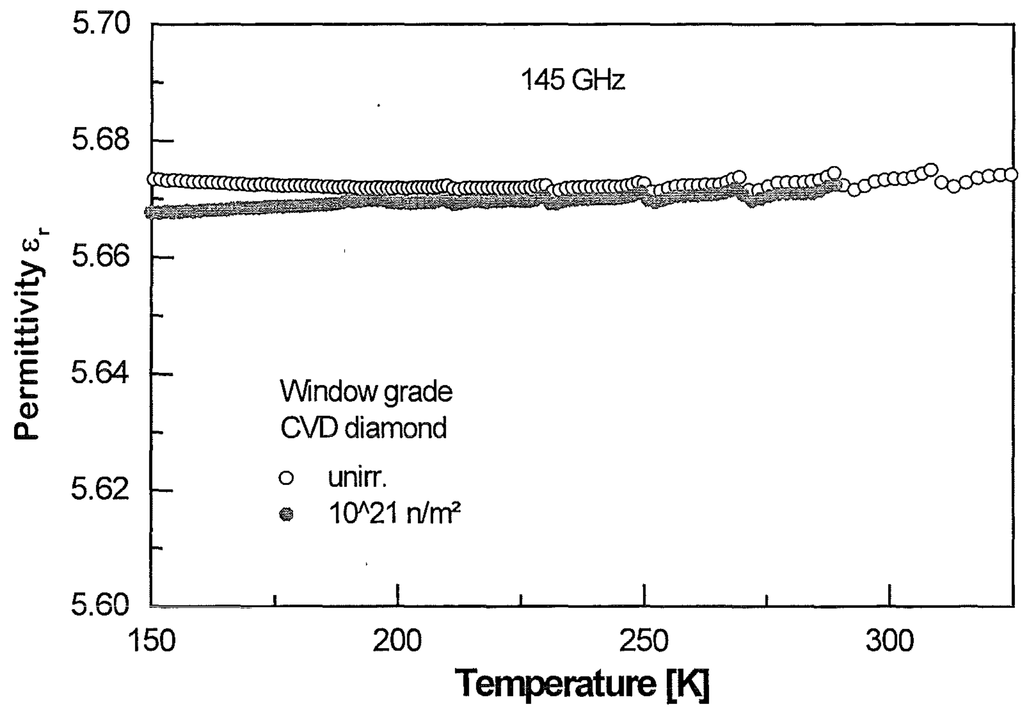


Fig.17b: The temperature dependence of the permittivity observed at 145 GHz in the window grade CVD diamond material before and after irradiation with fast neutrons.

6. Characterization of Window Materials (Subtask 2.6)

Within the scope of the previous task [3] and in Annex 2, it has been demonstrated that the first full size window disks with dielectric losses superior to Sapphire can be produced successfully. However, it became evident that inhomogeneities in mm-wave absorption along the disk axis, as well as in the radial direction, were of concern [4]. A typical example was the "FZK" disk which was produced by DeBeers to the size of 105 mm x 2.2 mm using the microwave plasma assisted CVD technique [5] where pronounced variations in the dielectric loss tangent were measured at 145 GHz, for example by line scans taken over two orthogonal diametral directions (Fig. 18). In the present task, window material from various sources were characterized aiming at further refined CVD diamond grades that should ideally combine good homogeneity with very low tanδ level.

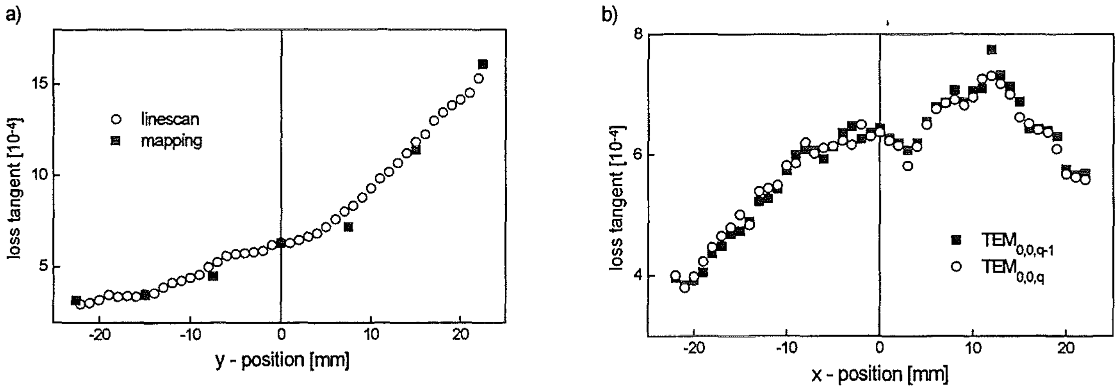


Fig. 18: Line scan of tanδ along the a) x-direction and b) y-direction in "FZK" disk at 145 GHz.

6.1 Window Materials from De Beers

The window disk studied at the beginning was produced to allow first brazing studies on low loss grades. The disk which received the name "Brazing" disk was 85mm in diameter and 1.6mm thick. The next one was produced to serve as "reference window type" with 4 inch diameter with actual geometries of 100 mm x 1.6 mm. It was given the internal name "Super-FZK", in continuation of a series of names starting with the "FZK" disk presented in [4]. The third one was produced to investigate and to demonstrate the growth process adopted for even more extended window geometries, such as for Brewster windows. This disk was denominated "Star of FZK", its dimensions are 119 mm x 2.2 mm.

The temperature dependent measurements between 70 K and 370 K showed for all three disks – like for the "FZK" disk - no significant changes of tanδ and ε_r with temperature. Whereas no difference occurred in the permittivity, which amounted to $\varepsilon_r = 5.67\ (\pm 0.01)$, striking reductions in tanδ of more than one order of magnitude could be observed for the three new disks. The increased scattering in loss tangent data for the "Super-FZK" window disk was due to the fact, that material came close to a perfect ultralow loss dielectric. Here the lowest loss tangent so far reported for a CVD diamond material at 145 GHz (at room temperature) was obtained with $\tan\delta = 6\ (\pm 2)\cdot 10^{-6}$. Even in the large "Star of FZK" losses hardly exceeded $2 \cdot 10^{-5}$ whereas the "Brazing" disk was in the 10^{-4} level (Fig. 19).

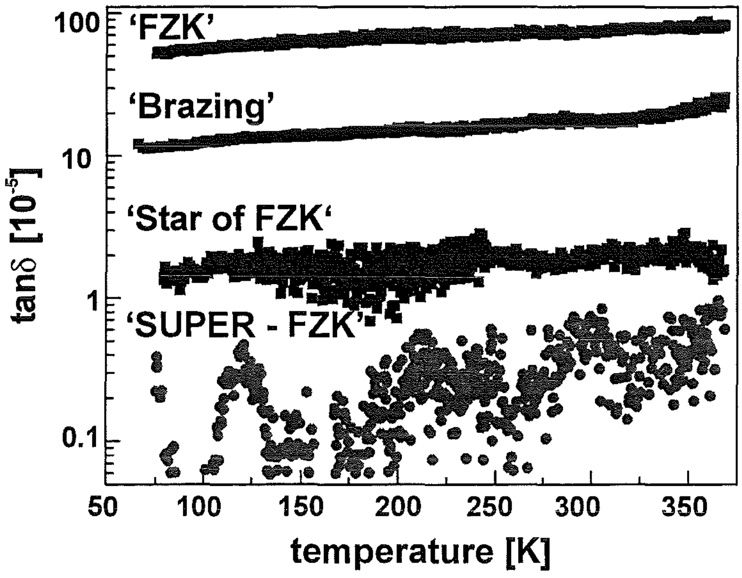


Fig. 19: Temperature dependence of tanδ at 145 GHz for the "FZK", "Brazing", "Star of FZK" and "Super-FZK" window disks.

The large disks could be used to determine the dielectric properties also at lower frequencies (10-20 GHz, 30–40 GHz) to cover a wider range for the experimental assessment of the frequency dependence. Typically a moderate decrease in frequency is observed which can be well described by an inverse square root law (tanδ ~ $f^{-1/2}$). At lower frequencies, inhomogeneities are especially critical because of the larger beam size in the measurement which increases the inspected area. Thus a local lossy area (see also Fig.21) is the origin for the elevated loss value observed around 15 GHz in the "Brazing" disk (Fig. 20 and Annex 3).

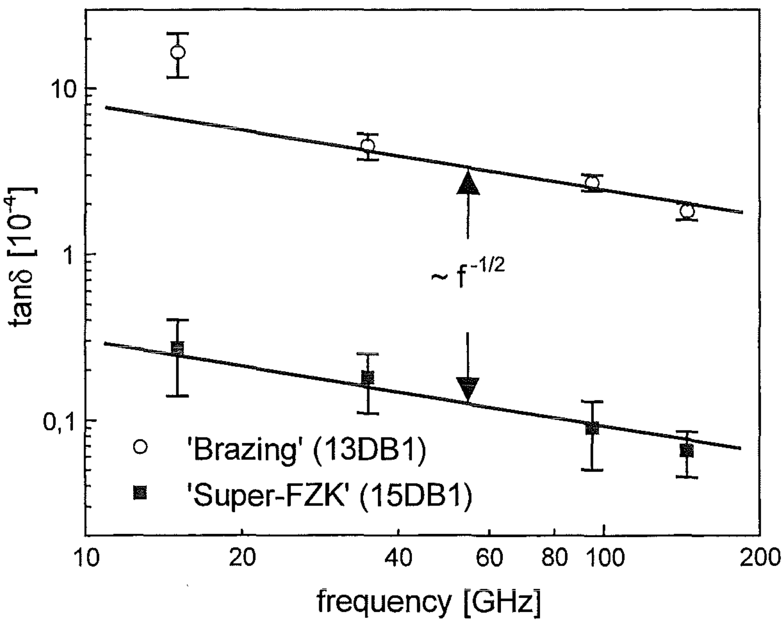


Fig. 20: Frequency dependence of tanδ of the locally inhomogeneous "Brazing"-window (13DB1) and the homogeneous "Super-FZK" - window (15DB1).

It is clear from [4] that different surface contributions to the loss typically arise at the nucleation and the growth face, which may be eliminated by polishing off an appropriate material quantity, but bulk losses could superimpose these. In the present new disks, no significant differences were observed along the growth axis by alternative placing the nucleation and the growth face into the resonator.

As for the distribution of the losses observed across the area of the disk, a new experimental set-up has been developed at 145 GHz based also on the open resonator technique (Annex 4) to determine a $\tan\delta$ mapping over almost the full area. The power of this technique is best exemplified by the $\tan\delta$ map obtained for the "Brazing" disk where a well localised region with elevated losses was observed in an off-centre position (Fig. 21). The same technique demonstrates the good homogeneity of losses in the "Super-FZK" window disk (Annex 4). The circles plotted in Fig. 22a do not represent the actual spot size of the measurement, but are demagnified for a clear lay-out, whereas for the actually mapped area (circle diameter = real spot size ~ 6 mm) is given in Fig. 22b. Obviously in the central region (about 60 mm in diameter), which is the most critical for a gyrotron window, $\tan\delta$ values did not go beyond the 10^{-5} level. A line scan across the "Star of FZK" window disk gives evidence of constant low loss area ($\tan\delta < 3 \cdot 10^{-5}$) in the inner area of at least 80 mm dia (Fig.23). The increase in the outer area may be enlarged to a certain extend by diffraction at the disk edges but has certainly a major material specific contribution.

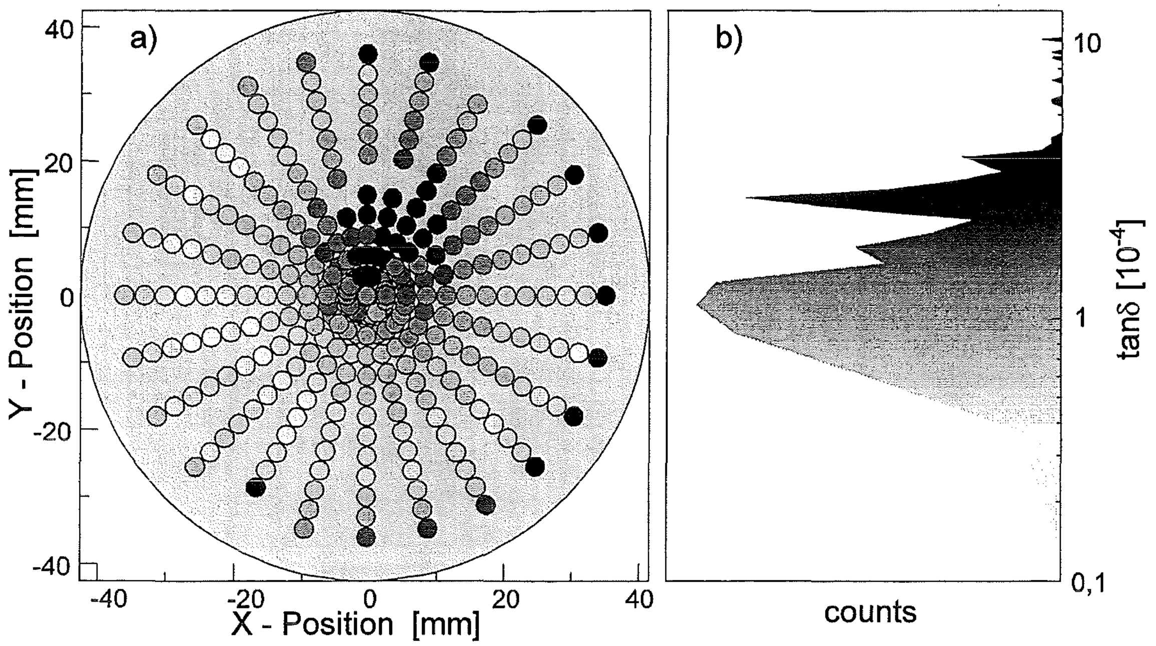


Fig. 21: Tanδ mapping for the "Brazing" window disk: a) demagnified spot size, b) distribution of measured tanδ points and their grey scale.

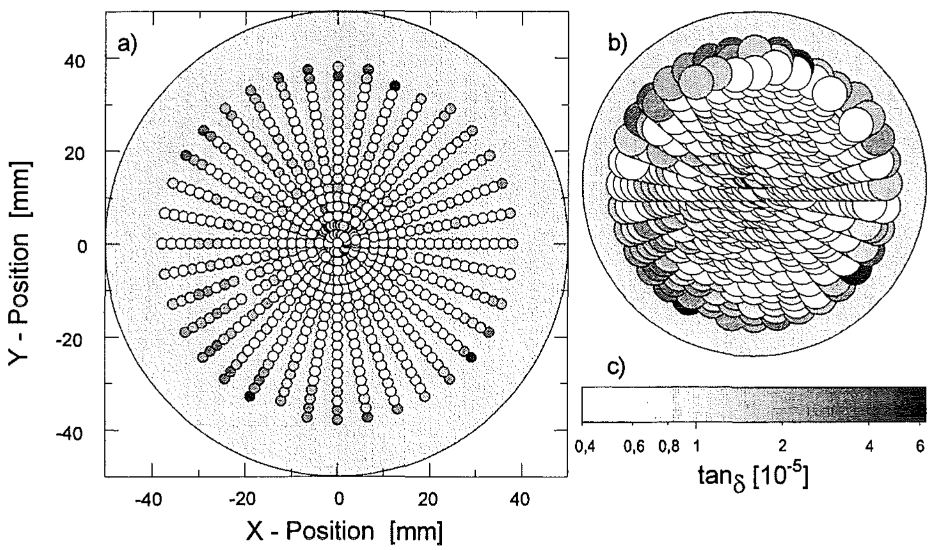


Fig. 22: Tanδ mapping for the "Super-FZK" window disk: a) demagnified spot size, b) actual spot size of 6mm and c) tanδ grey scale.

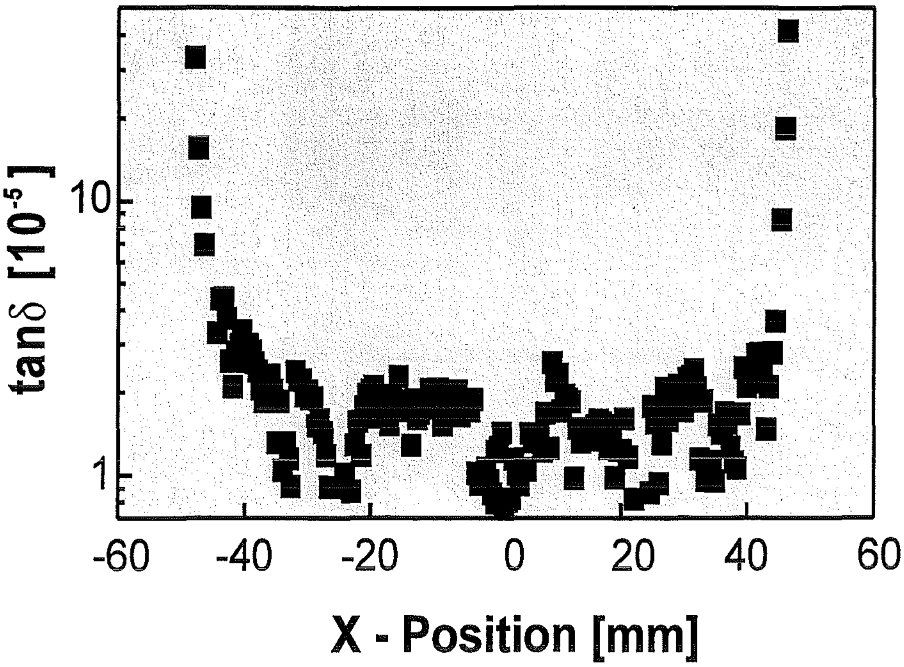


Fig. 23: Line scan across the diameter of the "Star of FZK" window disk.

6.2 Window Material from FhG-IAF

The free-standing diamond disks under investigation were prepared at the Fraunhofer Institute for Applied Solid State Physics (FhG-IAF, Freiburg, Germany) with another technique based on microwave-plasma-assisted chemical vapour deposition (MPA-CVD). However, the deposition was carried out in a novel microwave plasma system which uses an ellipsoidal cavity to generate very intense, spatially extended plasmas [6]. The performance of these microwave reactors is optimised with respect to long term stability and homogeneous growth on large areas. The present studies were performed on a set of different disks which were prepared by two versions of this CVD system: in a 2.45 GHz system powered with 6 kW microwave radiation disks with 2" and 3" diameter were prepared, whereas in a 915 MHz system equipped with a 60 kW microwave generator diamond disks with up to 6" in diameter can be produced. An unpolished free-standing high-grade diamond disk with 4 inch diameter and 1 mm thickness is exemplified in Fig. 24. Growth of similar disks with thickness up to 1.8 mm is demonstrated.

By using purified gases and optimising the process conditions, the quality and phase-purity of the CVD diamond disks could be brought to an excellent level. Samples prepared under optimum growth conditions exhibit properties approaching those of perfect type IIa diamond crystals. This includes an excellent broad-band optical transparency and a thermal conductivity exceeding 2000 W/m·K at room temperature [7].

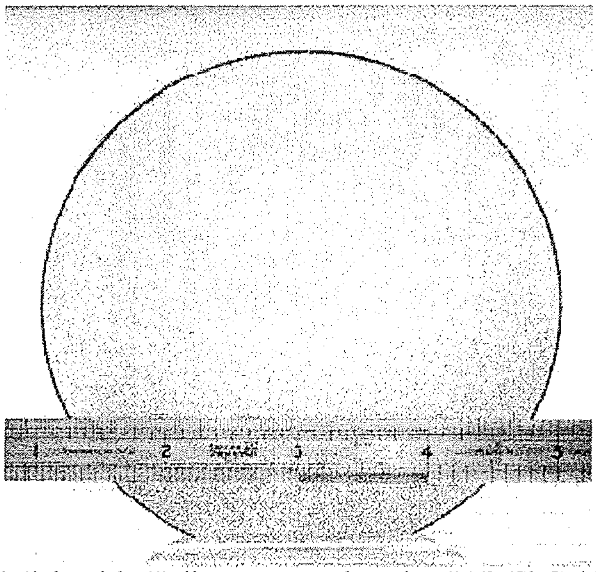


Fig. 24: A CVD diamond disk with 4" diameter produced at IAP-FhG (Annex 5).

At room temperature diamond wafers from different grades were tested for their dielectric loss. The test samples of 2" diameter were polished on growth surface and the measured loss values ranged from $2 \cdot 10^{-4}$ to $2 \cdot 10^{-2}$ at 30 - 40 GHz and from less than $5 \cdot 10^{-5}$ up to $1 \cdot 10^{-3}$ at 145 GHz (Tab. 3).

Table 3: Values of the loss tangent for different CVD diamond grades from FhG-IAP(Annex 5)

wafer	ERIII_14	ERIII_20	ERIII_49	ERVI_19
30 – 40 GHz	$2 \cdot 10^{-4}$	$35 \cdot 10^{-4}$	-	-
145 GHz	$0.4 \cdot 10^{-4}$	$3 \cdot 10^{-4}$	$5 \cdot 10^{-4}$	$< 0.5 \cdot 10^{-4}$

The permittivity was assessed to be 5.67 (±0.01) for all measurements at room temperature. Temperature dependent measurements were performed on the low loss specimen (ERIII_14). Down to the cryogenic regime, they were performed in two orientations of the specimen: one with the growth side and the other one with the nucleation side oriented into the resonator. Clear evidence for different surface contributions was observed (Fig. 25). As a consequence, in this particular disk a reduction of the loss level can be expected when removing high loss material from the 'as grown' nucleation side. The measurement of tanδ in the high temperature regime were performed in cooperation with the Russian ITER partners (IAP, Nizhny Novgorod (RF)). They did not give any evidence for additional thermally activated losses up to 225°C. Only for the upper temperature point of about 300°C , an increased tanδ level was apparent (Fig. 26 and Annex 3). Presently a high temperature measurement system is set up at the FZK-IMF I laboratories to obtain denser data set at 145 GHz up to 450°C.

At the present stage, it is demonstrated with this best disk that dielectric losses can be kept at a tanδ level below $5 \cdot 10^{-5}$ at 145 GHz (300 K). Taking into account that a further reduction of this value is possible by removing high loss material from the nucleation side, this technique has the potential to provide disks which are comparable to the CVD-diamond grades produced by DeBeers.

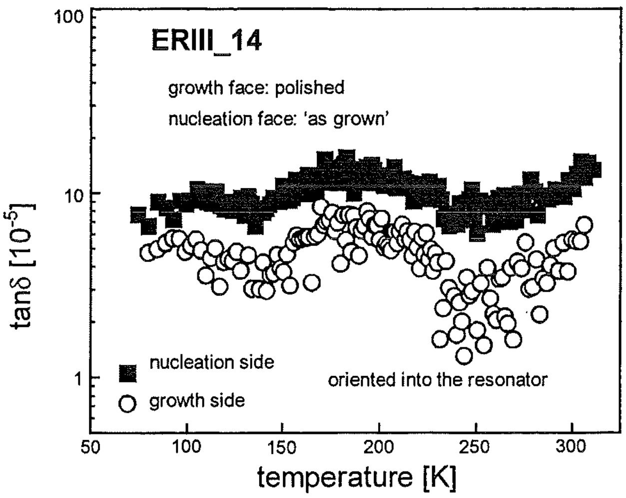


Fig. 25: Orientation dependent dielectric losses observed in CVD-diamond disk ERIII_14.

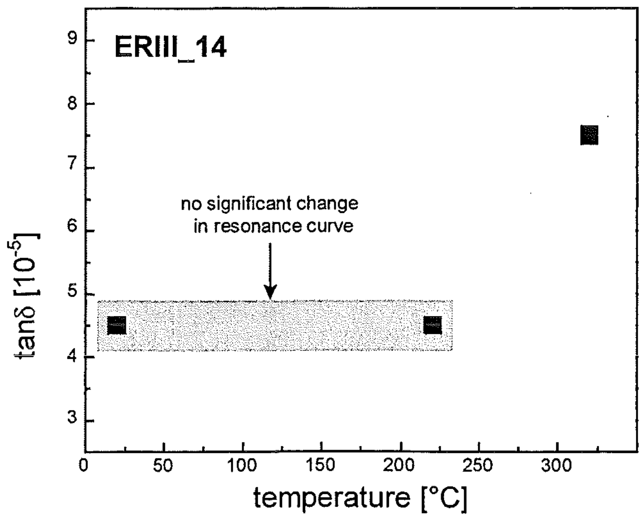


Fig. 26: Dielectric losses observed at 150 GHz and elevated temperatures in CVD-diamond disk ERIII_14 (Annex 3).

6.3 Window Materials from American Sources

In the recent year, continuous activities have been persued at the FZK to characterise window disks mainly of mainly European origin that were produced in the framework of the gyrotron development at JAERI (J) and MIT/CPI (USA). Typically, the task was to establish at one or at several selected positions of a large sized disk, the level of the dielectric loss tangent at 145 GHz. The main criterium for tanδ level to be kept at or below 10^{-4} could be generally confirmed.

In this cooperation framework, first disks from American sources of relevance for high power window applications came to the laboratory. A larger disk (63 mm diameter x 0.9 mm) could be inspected that was produced by Norton (Northboro, USA) by the DC Arc Jet method. The prohibitively large loss tangents above 10^{-3} that were measured at 145 GHz (Fig. 27) underline that this growing technique is at least at present not in a position to compete with material grown by the MPA-CVD techniques. A corresponding loss level was also found for a mounted structure with window disk of smaller size (~30 mm dia.) which was submitted to evaluate possible window development concepts at MIT/CPI. At the moment, material from other American sources with potentially lower loss levels is being under dielectric characterisation.

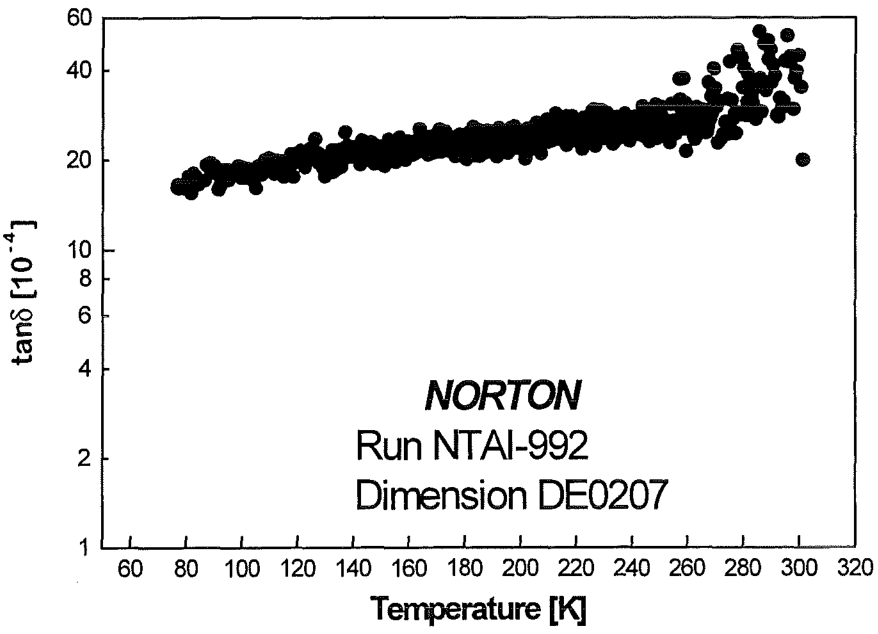


Fig. 27: Dielectric losses observed at 145 GHz in a CVD-diamond disk produced by the DC Arc Jet method.

6.4 Characterisation of Mechanical Strength in Window Grade Material

The mechanical strength properties, in particular the fracture strength, is a key material parameter that determines the resistance against thermal crack formation. Thermal crack formation limits the performance of a high power windows in absence of thermal run-away effects. So far, main considerations in CVD diamond window design have been laid on dielectric properties and thermal conductivity, because thermal gradients are generally low because of the outstanding thermal conductivity of the material. However, reduction of material performance by radiation damage and increasing challenges for transmitted power redefine the issue of mechanical strength. First preliminary studies on CVD window material in the ITER framework have been started, which take considerable profit from additional activities which are being followed on an European scope for establishing "Microwave and infrared industrial applications of Diamond". In the context of the latter project, powerful mechanical testing facilities based on the "ball on ring method" have been adopted for characterising CVD diamond and systematic studies have been performed on black and white CVD diamond grades grown by GEC Marconi (Caswell, UK) (Annex 6).

The mechanical tests within the ITER programme were performed on 8 specimens (12 mm diameter x 0.26 mm) of window grade material received from DeBeers. The strength data were obtained by transmitting the mechanical load onto the nucleation side of the disk via the ball, which means that the critical stress levels arise at the growth side which is oriented to the (support) ring of the test arrangement. The strength data obtained for the disks from both producers fit well into the general relationship between the thickness of the disk (inherently related to the size of the grains at the growth side) and the observed fracture strength (Figs. 28 and 29). This implies also that higher strength values can be expected when the nucleation side is under tensile stress (Fig. 30). Indeed, the European project with the GEC Marconi material has also demonstrated strength values up to 2000 MPa under these conditions (Annex 6). The scattering of the strength data for the first specimen set from DeBeers can be well compared to that determined on the sets from GEC Marconi. This means that remarkably high values of the Weibull modulus are obtained. A dedicated study on a black diamond grade has yielded a Weibull modulus of 22 which is much superior to the values determined in parallel for a high strength alumina grade ($m \approx 11$) (Annex 6). For an aperture diameter of 80 mm the required minimum disk thickness in dependence of the pressure difference is plotted in Fig. 31 [8].

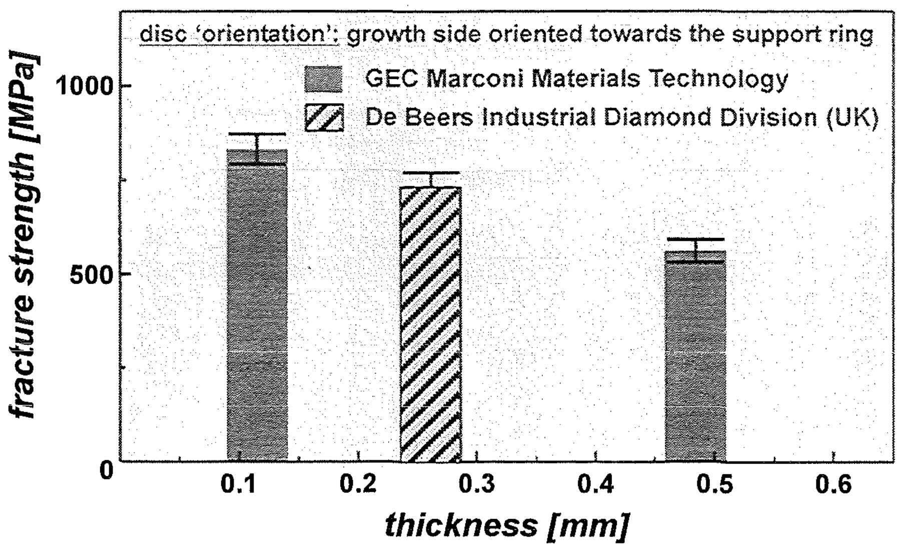


Fig. 28: The fracture strength of white (optical) CVD diamond grades tested with the growth side under tensile stress.

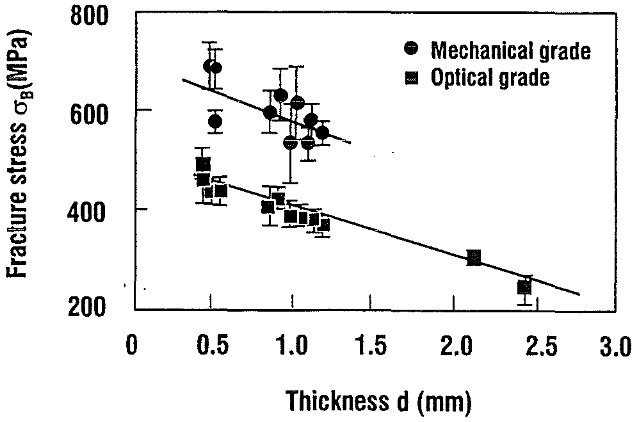


Fig. 29: Fracture strength of mechanical and optical CVD-diamond grades tested by DeBeers with growth surface under tensile stress [8].

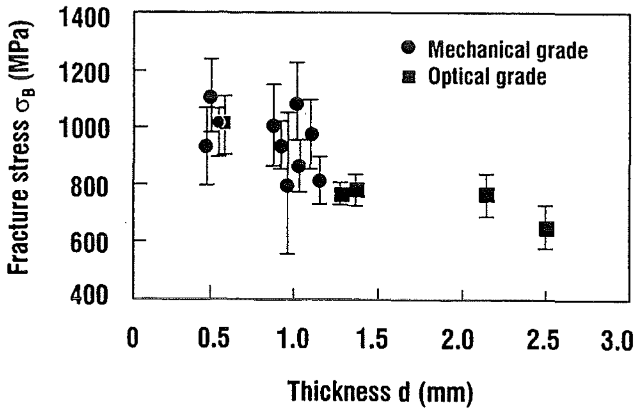


Fig. 30: Fracture strength of mechanical and optical CVD-diamond grades tested by DeBeers with nucleation surface under tensile stress [8].

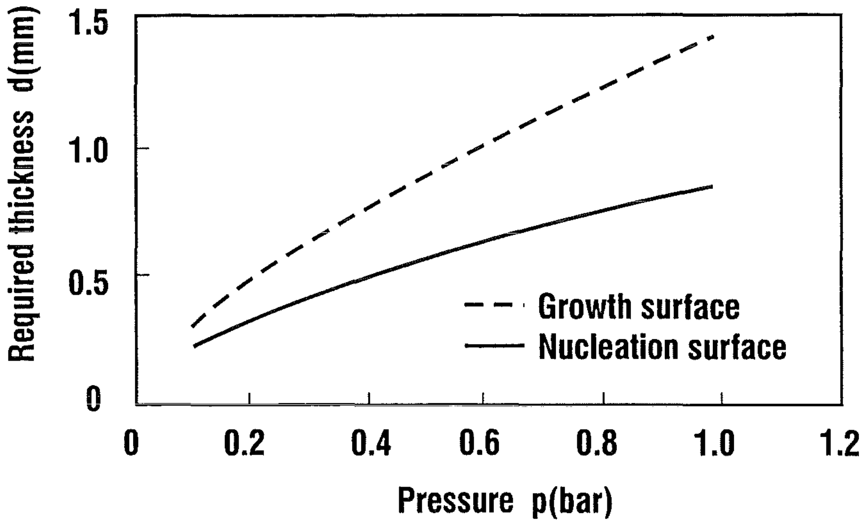


Fig. 31: Required minimum thickness of a CVD-diamond window with 80 mm aperture diameter and a safety factor of 4 in dependence of the pressure difference with growth or nucleation side under tensile stress respectively [8].

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List of Annexes

1 A. Kasugai, D.C. Ballington, A. Beale, J.R. Brandon, O. Braz, T. Kariya, K. Sakamoto, R.S. Sussmann, K. Takahashi, M. Tsuneoka, T. Imai, M. Thumm, 1998, "Chemical vapor deposition diamond window for high-power and long pulse millimeter wave transmission ", Review of Scientific Instruments, **69**, 2160-2165.

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Chemical vapor deposition diamond window for high-power and long pulse millimeter wave transmission

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To satisfy the electrical and thermomechanical requirements for a continuous wave millimeter wave beam transmission, a window assembly using a large size synthesized diamond disk has been developed. Such window systems are needed as a vacuum barrier and tritium shielding in future electron cyclotron heating systems for fusion plasma heating and noninductive electron cyclotron current drive. The diamond used in this study was manufactured by chemical vapor deposition (CVD) and consists of a polycrystalline diamond disk 96 mm in diameter and 2.23 mm thick. The disk was built into an assembly in which two Inconel tubes were bonded on both sides of the plate to provide vacuum shielding and water cooling to the edge of the disk, leaving an effective window aperture of 83 mm. It will be shown that, as a result of the high thermal conductivity and low dielectric loss exhibited by this grade of CVD diamond, the temperature increase of the window due to the absorption of high-power millimeter wave radiation could be minimized by simple water edge cooling at room temperature. During transmission of a focused Gaussian beam of 170 GHz, 110 kW, 10 s, the temperature increase at the center of the window reached a steady state condition at a value of approximately 40 K, in good agreement with calculated values. Water-edge-cooled CVD diamond windows promise to provide a practical technical solution for the transmission of continuous millimeter wave transmission in excess of 1 MW. © *1998 American Institute of Physics.* [S0034-6748(98)02805-6]

I. INTRODUCTION

Radio frequency (rf) wave heating systems in the 100 GHz range are the most promising candidates for fusion plasma heating and current drive in future fusion plasma reactors.[1] The greatest handicap of such systems up to now has been the absence of a vacuum barrier/tritium shielding window able to withstand continuous wave (cw) transmission of a millimeter wave beam in the megawatt range. They have to act as an output window for the rf source (gyrotron) as well as a millimeter wave beam inlet on the fusion reactor torus. In the planed International Thermonuclear Experimental Reactor (ITER) such systems will have to withstand transmission of a 1 MW cw millimeter wave beam at 170 GHz.

Up to now, several window concepts have been suggested and experimentally checked.[2] For example, face cooled sapphire double disk window,[3] cryogenic sapphire window,[4,5] boron nitride window,[6] distributed window,[7] Au-doped silicon window,[8] silicon nitride window.[9] The following several steps have been undertaken by the Japan Atomic Energy Research Institute (JAERI) towards the development of a high-power millimeter wave window. In a first step, a 110 GHz gyrotron was fitted with a double disk sapphire window.[10] Due to the insufficiency of the applied FC-75 surface cooling, the gyrotron performance was limited to 0.4 MW$-$4.0 s at 110 GHz due to high heat generation or permittivity change.[11,12] Even by applying a cryogenic edge cooling at 20 K its maximum capacity was reached at 0.4 MW$-$1 s at 110 GHz.[13,14] In addition to these limitations, it is not easy to handle cryogenic temperatures. The third stage of development was carried out on a 170 GHz gyrotron by using a double disk silicon nitride window.[15] The maximum power levels and pulse duration with this window concept was 0.175 MW$-$10 s and 0.52 MW$-$0.75 s at 170 GHz. However, the power and pulse length were limited by thermal runaway due to large rf power deposition.

The possibility of using chemical vapor deposition (CVD) diamond as a window material in high-power gyro-

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tron and millimeter wave transmission lines was first suggested after it was demonstrated that the dielectric loss tangent of CVD diamond could be reduced to values closes or below 7×10^{-5} in material of sufficiently high purity.[16] In addition to a low dielectric loss, diamond exhibits a number of other attributes that make this material an ideal option for windows required to transmit high-power radiation extending from the far infrared to the millimeter wave part of the spectrum.[16–20] Compared to other known materials, diamond is the hardest; has the highest room temperature thermal conductivity (five times that of copper) combined with one of the lowest thermal expansion coefficients. It has a wide optical transmission range (with very low values of bulk absorption), is radiation hard and is chemically inert to all acid or base reagents. Recent progress in the new synthesis technology of chemical vapor deposition has made it possible to manufacture very high quality CVD diamond optical components either as flat plate windows or three-dimensional shapes such as domes.[19,21] The ability to manufacture CVD diamond as a reliable and robust engineering material is opening the way to the use of this material for a range of technically demanding applications. The main challenge for the specific application of gyrotron windows has been to manufacture CVD diamond plates of diameter of at least 100 mm and thickness between 1.6 and 2.3 mm that exhibit consistent low values of dielectric loss. It was also necessary for this application to demonstrate the possibility of manufacturing a flange assembly that allowed a vacuum tight fit to the gyrotron tube and the possibility of water cooling. To achieve all these objectives a close collaboration was established among JAERI, the Forschungszentrum Karlsruhe (FZK), De Beers, and DIAMANX. This article reports on preliminary results of this development work in which two large size windows were delivered to JAERI. One of the windows was made into an assembly that allowed the vacuum-tight mounting of the window to the gyrotron tube. This structure left a free diamond edge for water cooling which allowed the characterization of the window performance at high powers. The window properties and flange structure are described in Sec. II. Section III shows the experimental setup employing a 170 GHz, high-power gyrotron of JAERI and the experimental result of the transmission of 170 GHz millimeter wave with 110 kW−10 s through CVD diamond window. In Sec. IV, thermal simulation of application to the rf output window for 1 MW-cw gyrotron and discussions are described.

II. CVD DIAMOND WINDOWS

A. CVD material

CVD diamond plates of high optical quality up to 100 mm in diameter and over 2 mm thick can be produced on a commercial basis at DIAMANX under the name of DIAFILM™. These plates can be cut and processed to achieve surfaces with precise optical tolerances in terms of flatness and parallelism. General properties of DIAFILM material have been described in previous publications.[16,18–20]

For the specific application of interest in this work, it was necessary to study the dielectric loss of windows manufactured under different process conditions in order to ascertain the degree of variability and uniformity achieved in large windows of diameters up to 100 mm. Since this study started over three years ago, a number of window samples were tested which exhibited values of dielectric loss tangent ranging from 70×10^{-5} to under 0.8×10^{-5}.[2,22] Two windows[23] have been manufactured for JAERI gyrotron window as described in Table I.

TABLE I. General outlines of the two CVD diamond windows used in this study. Disk A: medium quality disk. Disk B: good quality disk.

Window	Diameter (mm)	Thickness (mm)	$\tan\delta$ ($\times10^{-5}$)	Comments
Disk A	96.1	2.3 ± 0.02 (6 $\lambda g/2$ at 170 GHz)	13	Measured at the disk center at 170 GHz (JAERI)
Disk B	100	1.85 ± 0.02 (5 $\lambda g/2$ at 170 GHz)	2	Measured at the disk center at 170 GHz (JAERI)

For optical application in the visible and infrared part of the spectrum, where it is a requirement, it is possible to process the surfaces of CVD diamond windows to roughness better than 10 nm. For dielectric transmission applications, however, it is not necessary to achieve surfaces smoother than 200–300 nm. The surfaces of the two windows described above were processed to a surface roughness of 250 and 280 nm on the nucleation and growth side, respectively.

B. Configuration of an assembled CVD diamond window

One of the windows described in Table I (disk A), shown in Fig. 1(a), was used to fabricate an assembly to allow for the mounting of the window to the gyrotron tube. The assembly, shown in Fig. 1(b), consists of two Inconel tubes bonded on both sides of the CVD diamond plate, 2 mm from the edge, using a proprietary technology. The Inconel cylinders have small molybdenum retaining rings that help to balance possible thermally induced stresses in the assembly. This assembly left a usable aperture in the diamond window of 83 mm.

The CVD diamond window with the bonded Inconel cylinders was subsequently assembled into a water cooling housing, as shown in Fig. 1(c), in which a cooling jacket of stainless steel was welded at the edge of the Inconel cylinders. The cross section of the complete assembled structure is shown in Fig. 2. The cross-section area and the surface area of the diamond immersed in the water are 4.46×10^{-2} and 18.9 cm^2, respectively. The volume of the cooling channel around the disk is approximately 60 cm^3 with a typical cross section area of 2 cm^2 and two stainless-steel pipes of 1/2 in. diameter allow water to flow into the cooling channels. Cooling water with the temperature of about 20 °C can be supplied at a maximum flow rate of 18.3 ℓ/min by a water pump of 2 kgf/cm^2 in a closed loop. In order to minimize corrosion to metal parts, the water in the closed loop cooling system has been treated with a commercial corrosion inhibitor (cc. 15, Freeston Ltd.).

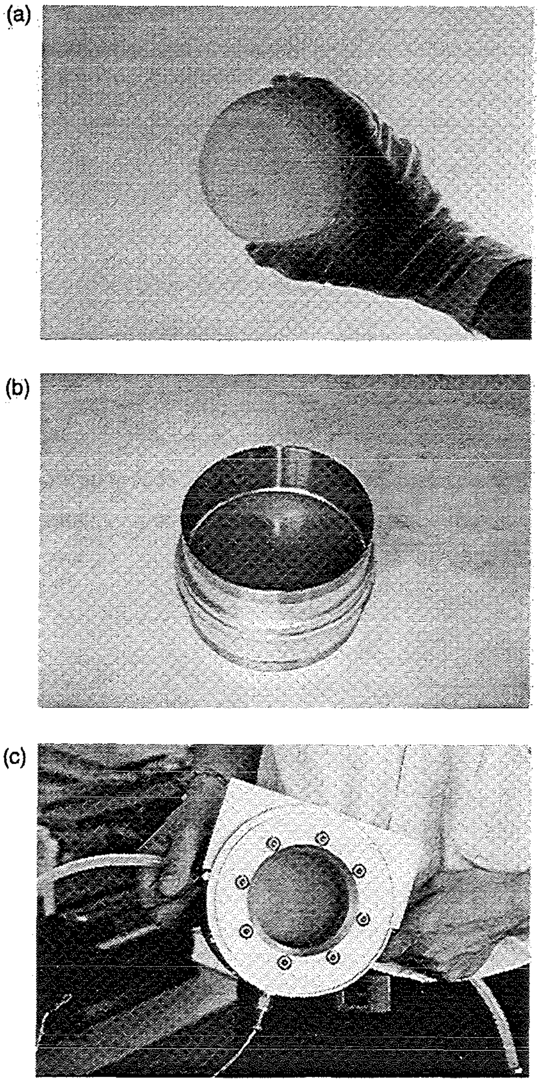

FIG. 1. Photographs of the several steps of development towards the CVD diamond window assembly, (a) CVD diamond disk before brazing, (b) metal bonded diamond disk with Inconel cylinders, (c) assembled diamond window with water cooling housing.

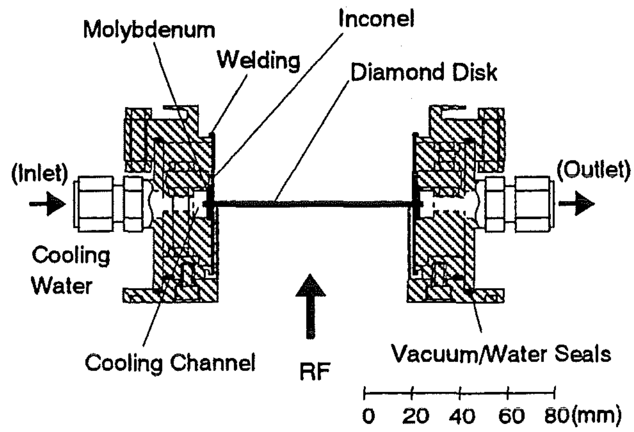

FIG. 2. Technical drawing of the cross section of the assembled diamond window.

III. HIGH POWER LONG PULSE EXPERIMENT

A. Configuration of experimental setup

Figure 3 shows a schematic drawing of the experimental setup. To be able to evacuate the rf guiding system the complete water-edge-cooled window assembly was placed into a test box inserted in a transmission line. The required rf power is provided by a 170 GHz JAERI/Toshiba gyrotron.[15] This microwave tube provides an rf output of $0.175\,\mathrm{MW}-10\,\mathrm{s}$ or $0.52\,\mathrm{MW}-0.75\,\mathrm{s}$ in operation using a depressed collector. The rf power from the gyrotron is introduced into the window test box via an evacuated corrugated HE11 waveguide with a diameter of 88.9 mm. The pressure in the transmission line, evacuated by two turbomolecular pumps, is under 10^{-2} Pa. In order to increase the power density and to reduce the spot size on the target disk, the waveguide was nonlinearly tapered down to the diameter of 31.75 mm. The rf beam transmission itself has been realized by launching the rf power from this open waveguide mouth and thus radiated as a Gaussian-like beam through the diamond window. The full width at half maximum (FWHM) of the rf beam at the position of the window disk has been determined to be about 23 mm which is much smaller than the aperture of the CVD diamond window system. The transmitted rf power is finally absorbed in a dummy load at the end of the transmission line. The rf power can be rerouted via a miter bend type waveguide switch into a calorimetric load to measure the power level of the incident rf.

A two-dimensional infrared (IR) camera has been used to determine the increase of center temperature of the CVD diamond window. A detecting wavelength of 3–5.4 μm has been chosen due to the nonpermeable spectrum in the wavelength range of only 3–5 μm.[17,18] The calibration of the temperature from the radiation spectrum observed by the IR camera was performed in advance by measuring the disk edge temperature by directly contacted thermocouples. The temperatures of the cooling water at the in- and outlet was measured by thermocouple. The arcing at the diamond window is monitored by photomultiplier tube to protect the diamond disk.

B. Experimental results

The rf power level for the entire series of experiments was fixed to 110 kW at the window test box while the pulse duration was gradually extended up to 10 s. The pulse dura-

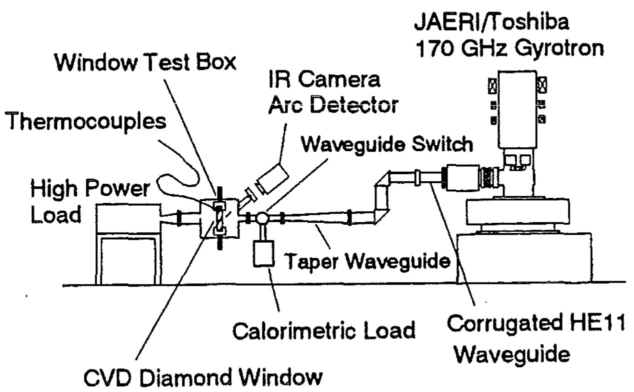

FIG. 3. Schematic drawing of the experimental setup using the JAERI gyrotron facilities (transmission power: 170 GHz, 110 kW, 10 s).

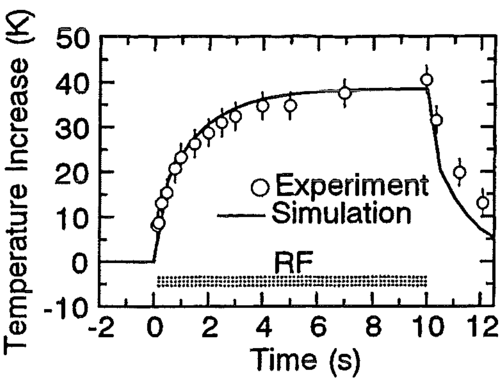

FIG. 4. Time behavior of the window center temperature increase for a 110 kW−10 s millimeter wave beam transmission. Open circles represent the experimental data and the solid curve shows the simulation result taking a heat transfer coefficient of 20 kW/(m^2 K) into account.

tion was limited by the capacity of the power supply system required for the gyrotron operation. The experimentally determined center temperature increase of the CVD diamond window during a 10 s long rf pulse is shown in Fig. 4. The applied flow rate of the cooling water for this measurement was 18.3 ℓ/min. As one can see the window center temperature increased from 23.4 to 64.5 °C and nearly reaches the steady state condition after 10 s. Also the temperature increase of the cooling water at the outlet was stabilized at $\Delta T = 0.3$ K after about 7.0 s. From these data the power deposition inside the diamond window was determined to be 380 W. The expected deposition power is given by the following equation:

$$P_{\text{dep}} = \frac{1}{c_0} \pi \cdot f (1+\epsilon') \tan \delta \cdot P_{\text{rf}} \cdot d, \tag{1}$$

where P_{dep} is the deposition power, c_0 the speed of light in vacuum, f the frequency of the incident rf, ϵ' the permittivity, tan δ the dielectric loss tangent, P_{rf} power of incident rf, and d the thickness of the disk. Applying the parameters of diamond window (disk A) shown in Table II, P_{dep} was estimated to be 380 W. The expected deposition power agreed very well with estimated deposition power from the temperature increase of the cooling water. During the full 10 s operations no arcing and no outgassing at the diamond window were observed and thus a very stable power transmission was confirmed.

The experimental data were compared to numerical simulation. To simulate the time behavior, the following one-dimensional radial heat flow equation with a convective condition, which is assumed an azimuthally symmetric distribution of the power deposition and heat flow, has been used:

$$\rho C \frac{\partial T}{\partial t} = \frac{K}{r} \frac{\partial}{\partial r} \left(r \frac{\partial T}{\partial r} \right) + Q, \tag{2}$$

$$Q = \pi f \epsilon_0 \left(\frac{1+\epsilon'}{2\sqrt{\epsilon'}} \right) \epsilon' \tan \delta |E(r,t)|^2, \tag{3}$$

$$q = \alpha_c (T - T_0), \tag{4}$$

TABLE II. Material properties of the CVD diamond window disks used for the simulations of the thermal behavior.

Parameters	Unit	Simulation 1 (Disk A)	Simulation 2 (Disk B)
Diameter of disk	mm	96.1	100
Thickness of disk	mm	2.23	1.853
Diameter of aperture	mm	83	83
Width of brazing	mm	3	3
Width of cooling zone	mm	2	4
Frequency	GHz	170	170
Loss tangent		1.3×10^{-4}	2.0×10^{-5}
permittivity		5.685	5.685
Thermal conductivity	W/(mK)	1800 (at room temp.)	1800 (at room temp.)
Mass density	kg/m^3	3.54×10^3	3.54×10^3
Specific heat	J/(kgK)	520 (at room temp.)	520 (at room temp.)
Heat transfer coefficient	kW/(m^2K)	20	20
Temp. of cooling Water	K	300	300
Input rf power	MW	0.5	1
RF profile		Flat (ϕ60 mm)	Gaussian (FWHM:33 mm)

where T is the window temperature expressed as $T(r,t)$ (r: radius, t: time), ρ (3.54×10^3 kg/m^3) is the mass density, C ($7.42\times10^{-2}T^{1.556}$ J/(kg K) in the range of $300\ \text{K} < T < 500$ K) is the specific heat, and K [1800 W/(mk) at 300 K] is the thermal conductivity. Q is the rate of power deposition. ϵ_0 is the dielectric constant in vacuum, and $E(r,t)$ the electric field strength of the incident rf beam. Equations (2) and (3) are solved using a one-dimensional finite element method with the boundary condition of Eq. (4). q is the heat flux on the cooling surface, α_c the heat transfer coefficient between the diamond disk and the cooling water, and T_0 the temperature of the cooling water. The solid line curve in Fig. 4 shows the results of the simulation using a loss tangent of 1.3×10^{-4}, an rf power of 110 kW and a pulse duration of 10 s. The best agreement was obtained by taking a heat transfer coefficient of $\alpha_c = 20$ kW/(m^2 K) into account.

Figure 5 shows the measured dependence of the window

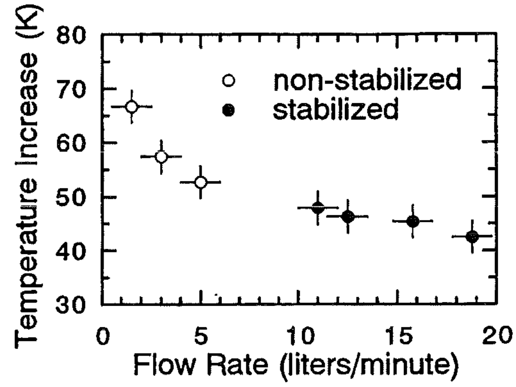

FIG. 5. Dependence of the window center temperature increase after 10 s (110 kW) operation on the flow rate of the cooling water. The closed circles represent stabilized temperature after 10 s, and the open circles represent nonstabilized temperature.

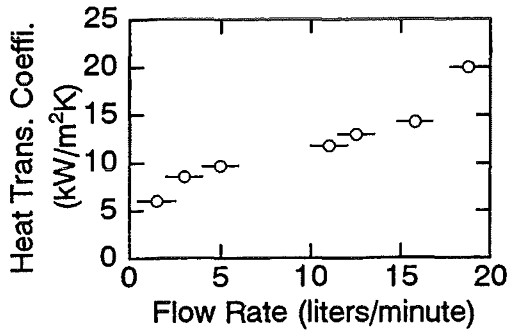

FIG. 6. Dependence of the heat transfer coefficient α_c between the diamond edge and the cooling water on the water flow rate. α_c is roughly proportional to flow rate.

center temperature increase on the cooling water flow rate after a 10 s rf transmission. The temperature increase was almost stabilized after 10 s for the flow rate over ~ 11 ℓ/min (closed circles). For smaller flow rates (open circles), stabilization could not be obtained. On the other hand, a further increase of the flow rate shows a much weaker influence on the temperature increase. By comparing the experimental results to the calculated values it was found that the heat transfer coefficient α_c, as shown in Fig. 6, is roughly proportional to the flow rate. As a result of these experiments it was found that in our configuration a minimum flow rate of ~ 11 ℓ/min corresponding to a $\alpha_c \sim 12$ kW/(m^2 K) had to be applied to achieve an effective cooling.

IV. DISCUSSIONS

The ability of a CVD diamond window with a simple water edge cooling to withstand the cw transmission of a millimeter wave beam with power levels up to the megawatt range has been demonstrated. Therefore a complete window assembly employing a metallized and bonded CVD diamond disk was placed in the output beam (Gaussian beam of FWHM $\sim$23 mm) of a 110 kW, 170 GHz 10 s gyrotron. The influence of varying the flow rate of the applied water edge cooling system on the heat transfer coefficient was investigated. Based on the experimental results, numerical simulations on the performance of CVD diamond window have been carried out for two types of diamond disks: The first one for the higher loss disk, which was used for this experiment (disk A) and the second one for a higher quality disk (disk B). The material properties and assumed rf beam parameters for these calculations are summarized in Table I and Table II. The results of the calculated dependence of the window center temperature increase versus time for both cases can be seen in Fig. 7. For case 1 [disk A ($\tan\delta = 1.3 \times 10^{-4}$)] a flattened beam profile with a power level of 0.5 MW was assumed. The geometry of the window aperture and cooling zone of the CVD diamond disk is the same as the one used in this high-power experiments. The calculated center and edge temperature increases stabilize at 150 and 70 K, respectively. It is obvious that such a window assembly easily can withstand the rf beam demands mentioned above. The same window assembly was assumed in the case of the temperature increase simulation of the disk B ($\tan\delta = 0.2 \times 10^{-4}$). In this case an incident rf power of 1 MW and a

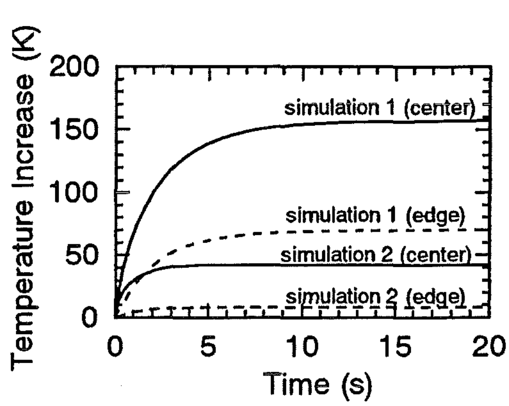

FIG. 7. Simulation results for two the types of window conditions. Simulation 1 shows the expected temperature increases vs time for the medium quality diamond disk at 0.5 MW, while simulation 2 shows those for the good quality disk at an rf power level of 1 MW.

Gaussian-like beam profile has been taken into account. In spite of the much higher rf demands, the center and edge temperature increases were stabilized at much lower values of 42 and 8 K, respectively. This can easily be explained by the lower loss tangent, longer cooling zone and thinner than disk A. Nevertheless, such a window would be able to handle a continuous wave transmission of a megawatt millimeter wave beam. The CVD diamond window will give a solution to the problem of a window which is capable of transmission of more than 1 MW and continuous wave at millimeter wave range and will enhance the progress especially of the high-power gyrotron and the electron cyclotron heating system.

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DEVELOPMENT OF OUTPUT WINDOWS FOR HIGH-POWER LONG-PULSE GYROTRONS AND EC WAVE APPLICATIONS

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Abstract-- **Electron cyclotron heating (ECH) is one of the main candidates for heating and current drive on ITER (170 GHz) and W7-X (140 GHz). High unit power (1 MW or greater) and high efficiency single-mode continuous-wave (CW) gyrotrons are under development in order to reduce significantly the systems costs. Face-cooled double-disk sapphire and silicon nitride windows (FC-75 liquid cooling), cryogenically edge-cooled single-disk sapphire (liquid nitrogen, liquid neon or liquid helium cooling) and silicon (230 K refrigerator cooling) windows, water-edge-cooled single-disk CVD-diamond windows and water-cooled distributed windows are being investigated in order to solve the window problem. A water-cooled window has two very important advantages; it employs a cheap and effective coolant and it is compact and probably more reliable than other solutions and thus can also be easily used as a torus window. The present paper summarizes the development status of high-power millimeter-wave windows with emphasis on CVD diamond.**

Keywords: **Gyrotron, Electron Cyclotron Heating, Dielectric Vacuum Window, CVD Diamond**

Introduction

ECH is one of the major candidates for heating, current drive (170 GHz, 50 MW, CW) and start-up (90 - 140 GHz, 3 MW, 5s) on the International Thermonuclear Experimental Reactor (ITER) tokamak [1-3] and will be the main start-up and heating scheme on the stellarator W7-X (140 GHz, 10 MW, CW) [2,3] at IPP Greifswald, Germany. ECH is extremely attractive from a fusion reactor engineering point of view, offering compact launch structures, high injected power density and a simple interface with the shield/blanket. Gyrotrons with an output power of at least 1 MW per unit are under development for economical use of such heating systems [3]. The requirement of CW operation results in extremely high demands on the material properties of the dielectric vacuum barrier windows that serve as both the primary tritium containment boundary at the torus and as the output windows of the gyrotrons. The former application is technically more demanding because a torus window must withstand a static 0.5 MPa pressure during off-normal events (safety requirement). It should use a fusion-reactor compatible cooling liquid and, in addition, its performance, both mechanical and millimeter (mm)-wave, must not be severely degraded by modest neutron and γ irradiation.

The most important aspect of high-power window development are the dielectric characteristics of the window materials i.e., loss factor $\tan\delta$ and permittivity ε'_r because they affect power absorption and reflection. The thickness d of a window disk is designed so that the power reflection is minimized:

$$d = N\lambda/(2\varepsilon'^{1/2}_r),$$

where N is an integer and λ is the free-space wavelength. It is evident that a temperature dependence of ε'_r complicates the choice of d and that once d is fixed, maximum transmission occurs at the series of frequencies f_i for which

$$N_i\lambda_i=2d\varepsilon'^{1/2}_r$$

(multi-passband window). Possible solutions for broadband windows are multi-layer windows (variation of permittivity for "anti-reflection coating") or "moth-eye-type" windows providing a tapering of the permittivity (Fig. 1). However, the ultimate solution for an ultra broadband window is the Brewster window. The Brewster angle for reflection-free broadband transmission of linearly polarized mm-waves through a window disk is given by

$$\theta_{Brewster} = \arctan \varepsilon'^{1/2}_r.$$

Types of High-Power Windows

Four general classes of high-power long-pulse capable windows are being developed (Fig.2): distributed, liquid-edge-cooled and gas-surface-cooled single-disk, and liquid-surface-cooled double-disk [4-7]. A variety of low loss-tangent dielectric materials (boron nitride, silicon nitride, sapphire, Au-doped silicon and MACVD (Microwave Assisted Chemical Vapor Deposition) diamond) are either currently used or under active development [4,7-11].

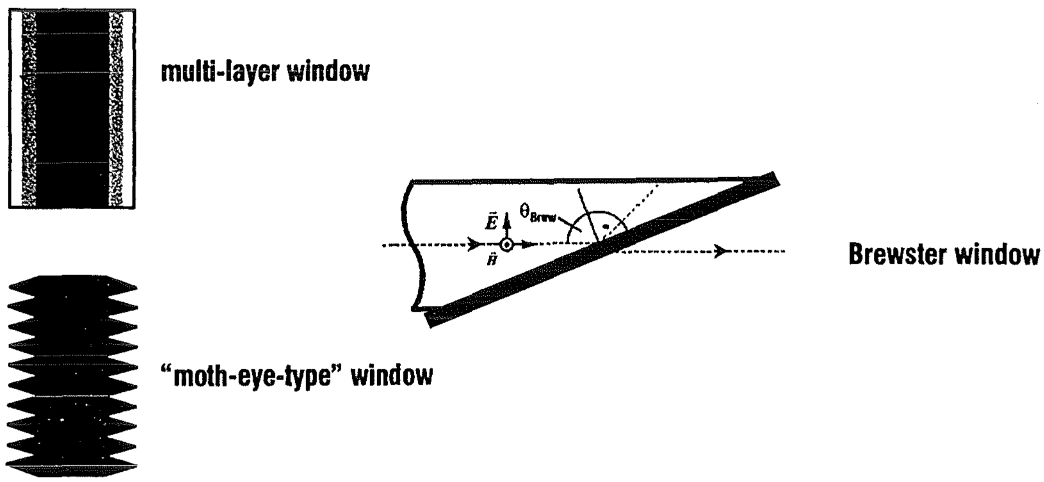

Fig. 1: Different solutions for broadband microwave windows.

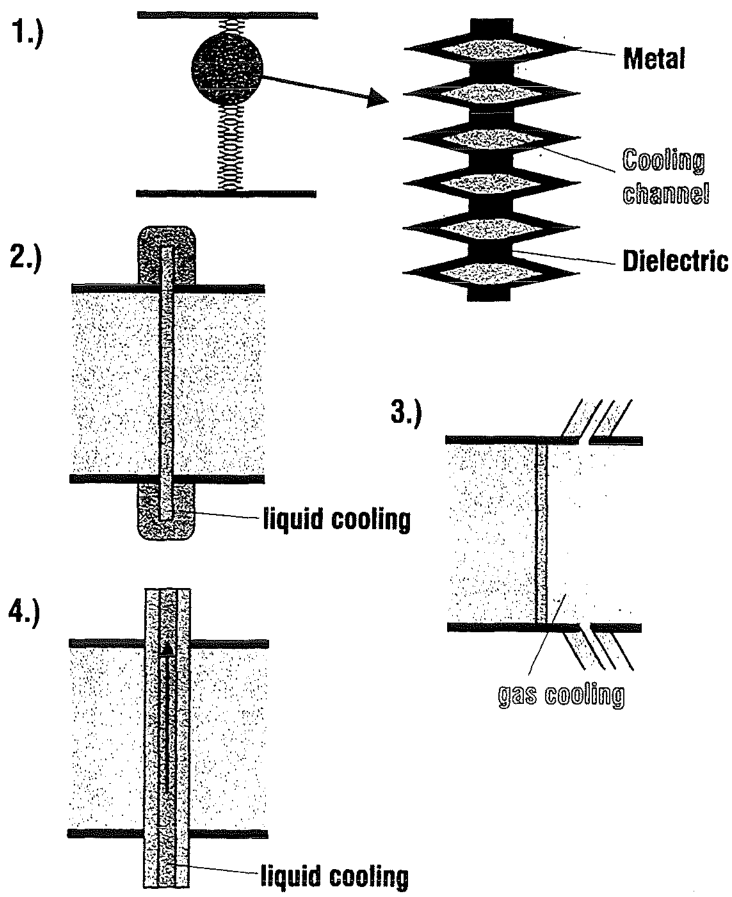

Fig. 2: General classes of high-power long-pulse capable microwave windows.

The distributed window consists of a planar slotted structure of alternating thin bars of dielectric material between microchannel cooled metal ribs [5]. Disadvantages are the complicated and expensive mechanical structure (large number of window elements), the high losses, the danger of arcing, even in an evacuated waveguide and its limitation to only one direction of polarization. Cryogenically edge-cooled single-disk windows are an attractive option [9-11]. For a number of materials including sapphire, it is found that the thermal conductivity increases and the loss tangent decreases as the temperature is reduced. Operating points are usually found between LHe and LN_2 temperatures, depending on the material. The advantages of this type of windows are that they are low loss and generally not polarization dependent. Disadvantages are that they are large (use of cryogens implies dewars and insulations surrounding them), the operating point is subject to thermal runaway and they must be prevented by a cold trap from cryo-pumping of the dust in the antenna waveguide. Liquid-surface-cooled double-disk windows have the disadvantages that two disks are required per barrier, instead of one as in the above concepts, and the use of dielectric fluids whose properties generally conflict with safety requirements (free fluorine readily reacts with tritium, decanes are flammable, etc.). Use as a torus window is thus probably excluded.

Several methods exist for increasing the power capability of the window without fundamentally altering the design [4]. One is by optimizing the beam profile. Flat, non-Gaussian profiles increase the power handling capability by as much as 50 %, and annular profiles by as much as 100 %. The disadvantage of these methods is that they require the use of waveguide mode converters or mode-converting (non-quadratic profiled) mirrors. Alternatively, high-aspect-ratio elliptical (or rectangular) windows can be used to reduce the thermal path length in one dimension. However, unless the transmission remains in elliptical (or rectangular) waveguide, rather lengthy mode converters or profile-transforming mirrors are required.

Present State-of-the-Art

The present experimental development status of high-power mm-wave windows is summarized in Table 1. As can be seen, no windows have been tested up to now at the MW power level in CW operation.

Material Selection and Options for 1 MW, CW

In order to define the appropriate concepts for the development of 1 MW, CW mm-wave windows one has to compare the thermophysical, mechanical and dielectrical parameters of possible window materials related to the load-failure resistance R' and the power-transmission capacity P_T at different temperatures [4].

Material	Type	Power (kW)	Frequency (GHz)	Pulse Length (s)	Institution
water-free fused silica	single disk inertially cooled	200	60	5.0	UKAEA/Culham
boron nitride	single disk water edge cooled	930	110	2.0	GYCOM-M
		330	110	10.0	GYCOM-M
		550	140	3.0	GYCOM-M
silicon nitride	single disk gas face and water edge cooled	130	84	30.0	NIFS/CPI
sapphire	single disk LN_2 edge cooled	530	118	5.0	CEA/CRPP/FZK/TTE
		285*	140	3.0	IAP/INFK
		500	140	0.5	FZK/IAP/IPF/IPP
		370	140	1.3	FZK/IAP/IPF/IPP
sapphire	single disk LHe edge cooled	410	110	1.0	JAERI/TOSHIBA
		500	110	0.5	JAERI/GA
sapphire	double disk FC75 face cooled	200	60	CW	CPI
		400	84	10.5	NIFS/CPI
		350	110	10.0	CPI
		350	110	5.0	JAERI/TOSHIBA
		200	140	CW	CPI
		500	170	0.6	JAERI/TOSHIBA
sapphire	distributed water cooled	65**	110	0.3	GA/JAERI
		200*	110	0.7	GA/CPI
diamond	single disk water edge cooled	300**	110	1	CPI/FOM
		50	110	CW	CPI/FOM
		450	110	2	GYCOM-M/GA
		110	170	10	JAERI/FZK

Table 1: Experimental parameters of present high-power mm-wave vacuum windows [3]. Note:* and ** indicates that the power corresponds to that of a 1 MW (*) and 0.8 MW(**) HE_{11} mode.

The features of boron nitride, silicon nitride (Kyocera SN-287), sapphire, Au-doped silicon and CVD diamond at room temperature and of sapphire, Au-doped silicon and CVD diamond at cryo-temperatures are summarized in Tables 2 and 3, where $R' = k \cdot \sigma_B \cdot (1-\nu)/E{\cdot}\alpha$
and $P_T = R' \rho \cdot c_p/((1 + \varepsilon_r') \cdot \tan\delta)$.

The LN_2-edge-cooled sapphire window of the 118 GHz TTE gyrotron (0.5 MW, 210 s), that operates close to the allowable lower limits of these two parameters, has R'=130 and P_T=80 [11].

The comparison of R' and P_T for the three materials BN, Si_3N_4 and sapphire clearly shows that there is no chance to use these dielectrics in an edge-cooled, single-disk window at room temperatures. Experiments at CPI in the US and at NIFS and JAERI in JA confirmed, that even a double-disk FC75-face-cooled sapphire window has a CW-power limit around 0.3-0.4 MW.

Using the available material parameters and employing various beam profiles, finite element computations revealed the options for 170 GHz, 1 MW, CW operation given in Table 4 [4]. Options 1 to 3 being water cooled, are preferred for their simplicity, in particular for use as a torus window. Irradiation tests with

Material	BN (CVD) p.c.	Si_3N_4 comp. (SN-287)	Sapphire (Al_2O_3) s.c.	Silicon Au-doped s.c.	Diamond (MACVD) p.c.
Thermal Conductivity k [W/mK]	50	59	40	150	2000
Ultimate Bending Strength σ_B[MPa]	80	800	410	3000	600
Poissons Number ν	0.25	0.28	0.22	0.1	0.1
Density ρ [g/cm³]	2.3	3.4	4.0	2.3	3.5
Specific Heat Capacity c_p [J/g K]	0.8	0.6	0.8	0.7	0.5
Young's Modulus E [GPa]	70	320	385	190	1050
Therm. Expans. Coeff. α [10^{-6}/K]	3	2.4	5.5	2.5	1.2
Permittivity (145 GHz) ε_r'	4.7	7.84	9.4	11.7	5.67
Loss Tangent (145 GHz) $\tan\delta$ [10^{-5}]	115	30	20	0.35	1
Metallizing/Brazing Bakeout	o.k.	o.k. 550°C	o.k. 550°C	o.k 550°C	o.k. 500°C vacuum
Possible Size ∅ [mm]	145	400	270	127	160
Cost	medium	high	high	low	very high
Failure Resistance R' $R' = k\sigma_B(1-\nu)/E\alpha$	14.2	44.5	6.0	852	858
RF-Power Capacity P_T $P_T = R'\rho\, c_p/((1+\varepsilon_r')\tan\delta)$	0.04	0.36	0.09	318	225
Radiation Sensitivity n(10^{20}-10^{21}n/m²) γ/X (0.75 Gy/s)			no no	no no	no no

Tab. 2: Thermophysical, mechanical and dielectrical parameters of window materials related to thermal load -failure resistance and power transmission capacity of edge-cooled windows at room temperature (p.c. = poly-crystalline, s.c. = single-crystalline).

Material	Sapphire (Al_2O_3) s.c.	Silicon Au-doped s.c.	Diamond (MACVD) p.c.
Thermal Conductivity k [W/mK]	900 (20000)	1300	10000
Ultimate Bending Strength σ_B[MPa]	410	3000	600
Poissons Number ν	0.22	0.1	0.1
Density ρ [g/cm³]	4.0	2.3	3.5
Specific Heat Capacity c_p [J/g K]	0.8	0.7	0.5
Young's Modulus E [GPa]	402 (405)	190	1050
Therm. Expans. Coeff. α [10^{-6}/K]	5.5	2.5	1.2
Permittivity (145 GHz) ε_r'	9.3	11.5	5.63
Loss Tangent (145 GHz) $\tan\delta$ [10^{-5}]	0.57 (0.2)	0.35	1
Metallizing/Brazing Bakeout	o.k. 550°C	o.k 550°C	o.k. 500°C vacuum
Possible Size ∅ [mm]	270	127	160
Cost	high	low	very high
Failure Resistance R' $R' = k\sigma_B(1-\nu)/E\alpha$	130 (2871)	7389	4286
RF-Power Capacity P_T $P_T = R'\rho\, c_P/((1+\varepsilon_r')\tan\delta)$	71 (4460)	2719	1132
Radiation Sensitivity n($0.3 \cdot 10^{21}$n/m²) γ/X (0.75 Gy/s)	no no	no	no

Tab. 3 : Thermophysical, mechanical and dielectrical parameters of window materials related to thermal load -failure resistance and power transmission capacity of edge-cooled windows at LN_2-temperature 77 K (LNe-temperature 30 K) (p.c. = poly-crystalline, s.c. = single-crystalline).

neutrons ($3 \cdot 10^{20}$-10^{21} n/m²) and γ-rays (0.75 Gy/s) show no change of the loss tangent and permittivity of CVD diamond, sapphire and high-resistivity (HR) silicon [4].

The ITER partners distributed the R&D on 1 MW, CW ECH windows between themselves in the following way:
(a) water-cooled distributed sapphire window (US)
(b) water-edge-cooled diamond window (EU,JA)
(c) 230 K-cryo-cooled Au-doped silicon window (RF)
(d) LN_2-, LNe- or LHe-cryo-cooled sapphire window (EU,JA,RF).

	Material	Type	RF-Profile	Cross-Section	Cooling
①	Sapphire/ Metal	distributed	flattened Gaussian	rectangular ($100 \times 100\ mm^2$)	internally water cooled (300 K) $\tan\delta = 2\text{-}5 \cdot 10^{-4}$, $k = 40$ W/mK
②	Diamond	single disk	Gaussian	circular (∅ = 80 mm)	water edge cooled (300 K) $\tan\delta = 2 \cdot 10^{-5}$, $k = 1900$ W/mK
③	Diamond	Brewster	Gaussian	elliptical ($152 \times 63.5\ mm^2$)	water edge cooled (300 K) $\tan\delta = 2 \cdot 10^{-5}$, $k = 1900$ W/mK
④	Silicon Au-dop.	single disk	Gaussian	circular (∅ = 80 mm)	edge cooled (230 K), $\tan\delta = 2.5 \cdot 10^{-6}$, $k = 230$ W/mK
⑤	Silicon Au-dop.	single disk	Gaussian	circular (∅ = 80 mm)	LN_2 edge cooled (77 K) $\tan\delta = 4 \cdot 10^{-6}$, $k = 1500$ W/mK
⑥	Sapphire	single disk	flattened Gaussian	elliptical ($285 \times 35\ mm^2$)	LN_2 edge cooled (77 K) $\tan\delta = 6.7 \cdot 10^{-6}$, $k = 1000$ W/mK
⑦	Sapphire	single disk	Gaussian	circular (∅ = 80 mm)	LNe or LHe edge cooled (27 K) $\tan\delta = 1.9 \cdot 10^{-6}$, $k = 2000$ W/mK

Note that the power capability of options ②,③,⑤ and ⑦ is even 2 MW.

Table 4: Options for 1 MW, CW, 170 GHz ECH Windows [3]

CVD-Diamond Windows

As a potential new material for simple water-edge-cooled single-disk windows, diamond is attractive due to its good mechanical properties, modest dielectric constant, relatively low loss and excellent thermal conductivity. The temperature dependence of the loss tangent of CVD-diamond (DeBeers, diameter = 40 mm, thickness = 1.1 mm) at 145 GHz is presented in Fig. 3 together with sapphire (HEMEX grade) and Au-doped HR-silicon. Fig. 4 shows the temperature dependence of the permittivity of diamond compared to sapphire at 145 GHz. Current CVD capabilities have allowed for tests with diamond disks of up to 100 mm diameter and 2.5 mm thickness. In the temperature range 200-370 K the loss tangent and the permittivity of diamond are practically constant [12]. This is not the case for sapphire and silicon. The frequency dependence of the loss tangent of sapphire, diamond and silicon is proportional to f, $1/f^{1/2}$ and 1/f, so that the absorbed power is proportional to f^2, $f^{1/2}$ and a constant, respectively. The loss tangent values of the large diameter CVD-diamond disks are approximately 10^{-4} ($5 \cdot 10^{-5}$ for 0.6 mm thickness) with tendency to decrease, so that we consider a value of around $2 \cdot 10^{-5}$ as feasible [12]. Manufacturers (DeBeers) claim that they also can produce disks with up to 160 mm diameter which could be used as Brewster windows.

In order to validate the low-power loss tangent measurements on large-size CVD-diamond disks, a series of experiments using a 170 GHz, 0.2 MW, 10 s

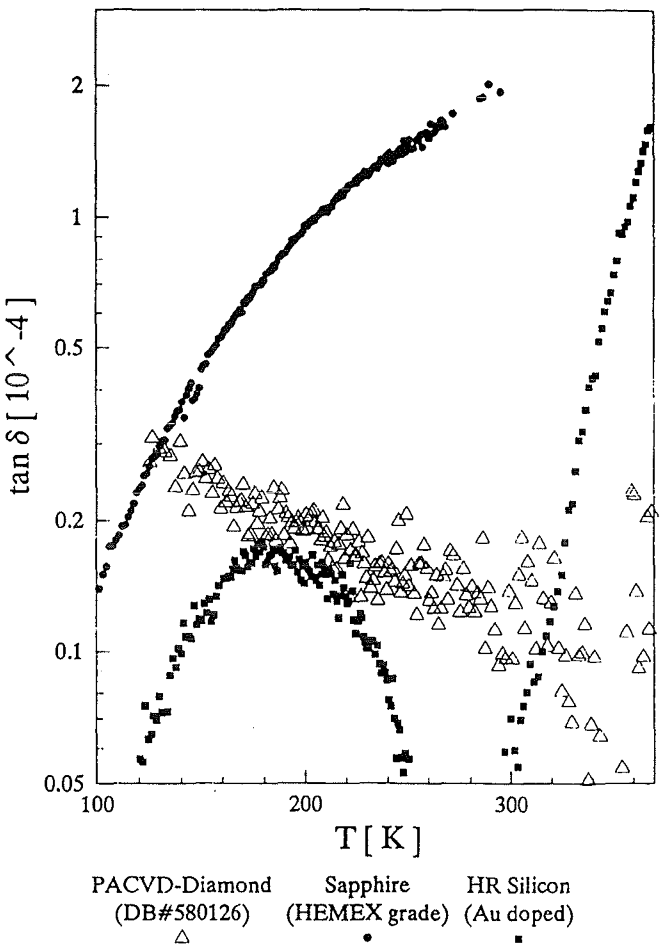

Fig. 3: Temperature dependence of tanδ at 145 GHz [3].

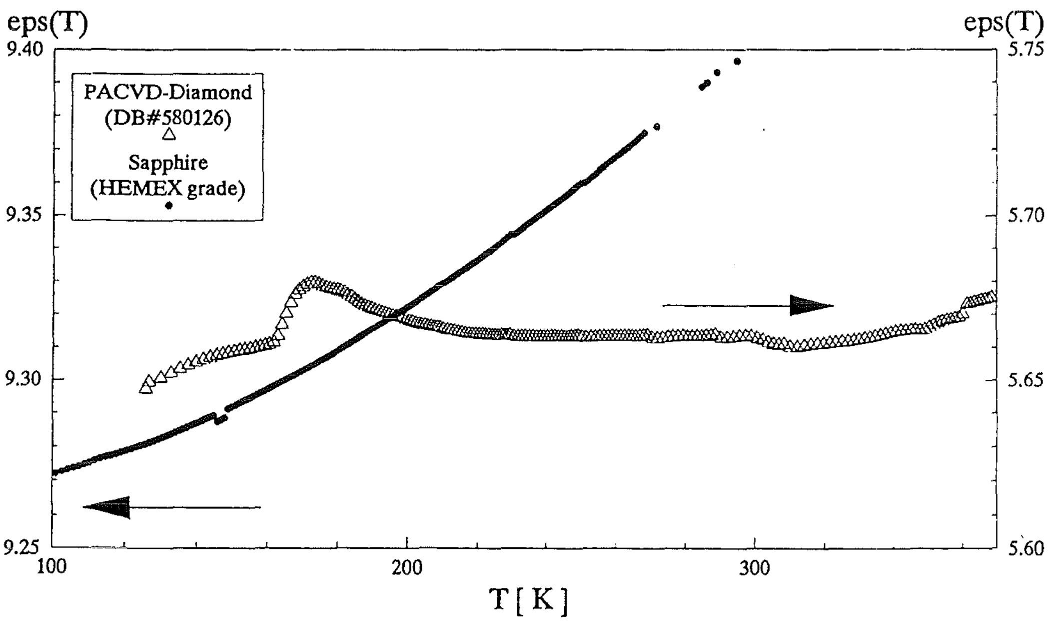

Fig. 4.: Temperature dependence of permittivity of CVD diamond compared to sapphire at 145 GHz.

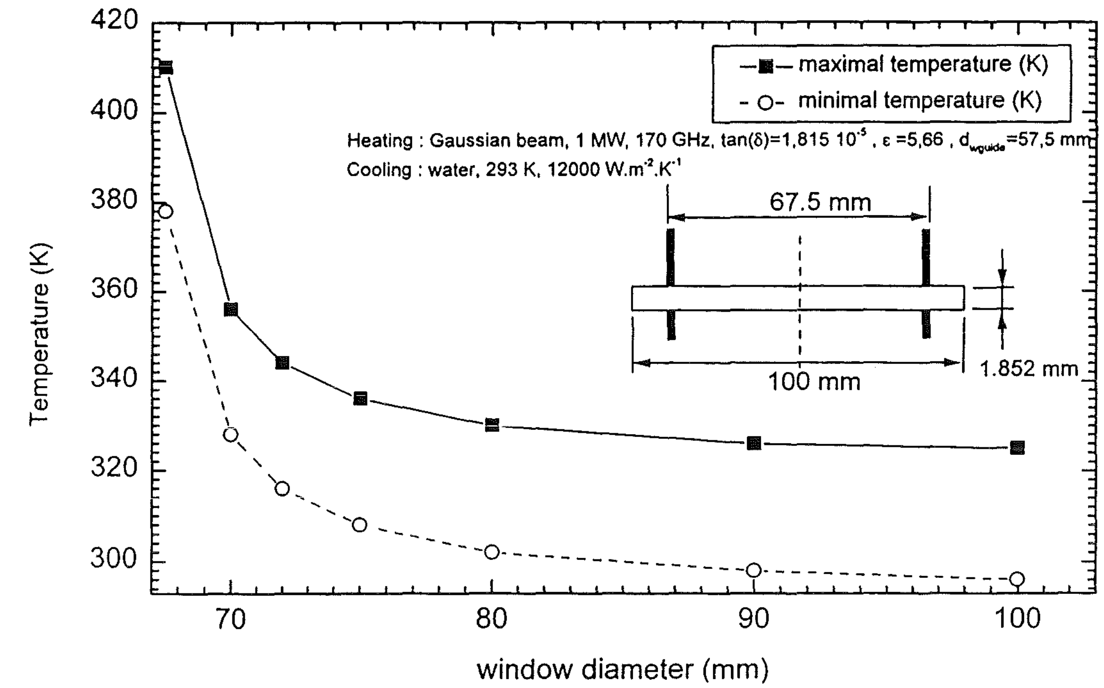

Fig. 5: Dependence of center and edge temperature of CVD-diamond windows on the width of the applied cooling rim at 1 MW mm-wave power (170 GHz).

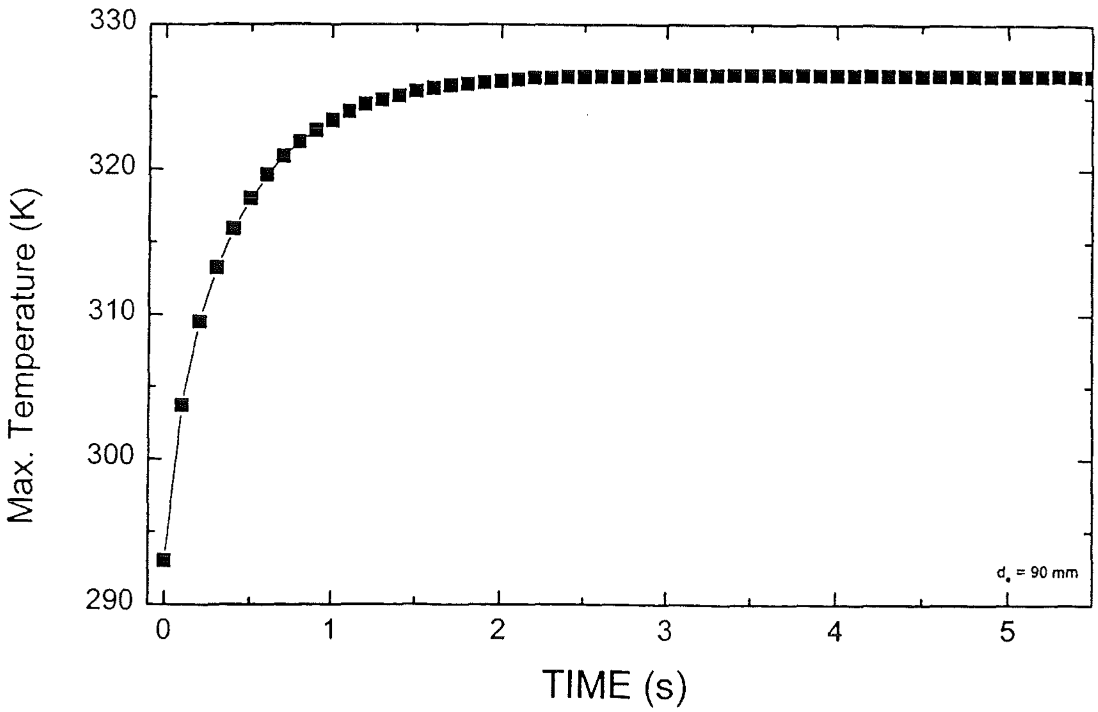

Fig. 6: Time dependence of center and edge temperature of CVD-diamond window (outer disk diameter = 90 mm) at 1 MW (170 GHz).

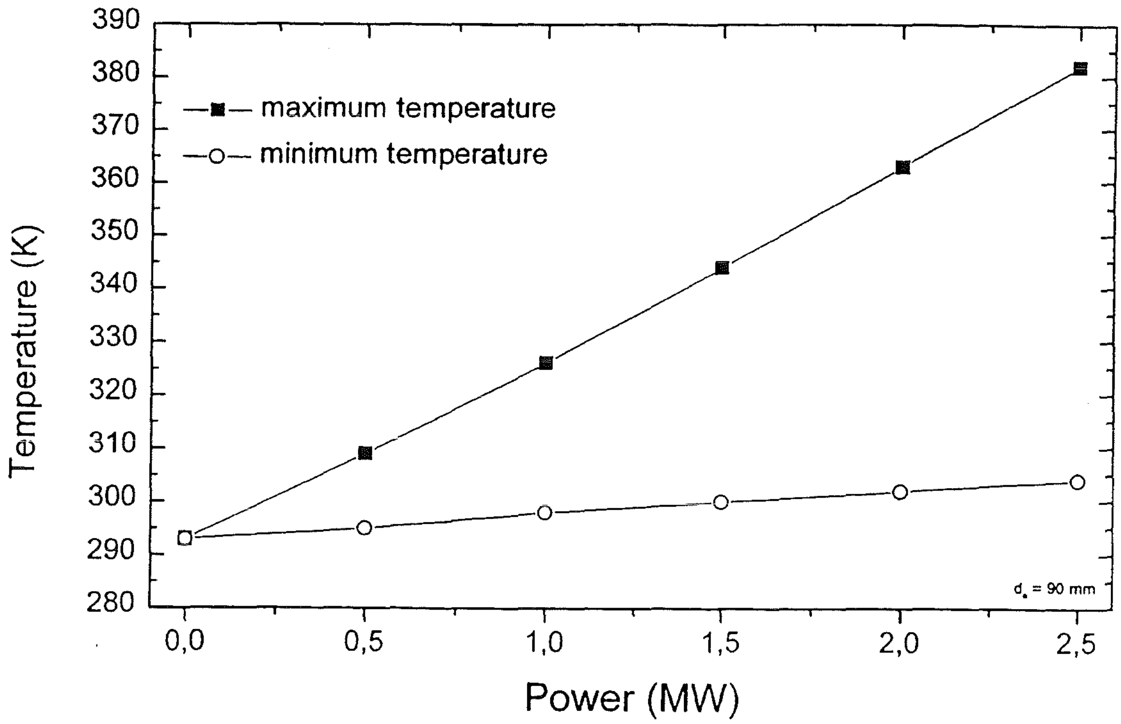

Fig. 7: Dependence of center and edge temperature of CVD-diamond window (outer disk diameter = 90 mm) on the transmitted mm-wave power (170 GHz).

JAERI/Toshiba gyrotron have been performed [13]. The dielectric loss tangent has been determined to be $\tan\delta=1.3\cdot10^{-4}$ which is in good agreement with the low power value. By comparing the experimental results with numerical simulations the thermal conductivity was estimated to be about $k \approx 1800$ W/mK.

Finite element calculations showed that the peak temperatures for CVD-diamond disks with a wide range of $\tan\delta$ and ratio of window aperture to disk diameter are acceptable (central temperature below 300°C). The window disk in a corrugated HE_{11} waveguide with 57.5 mm inner diameter should have an outer diameter of approximately 85-90 mm (Fig. 5). The corresponding time dependence of the center temperature is plotted in Fig. 6. The power absorbed in the disk is 400 W. Figure 7 presents the maximum and minimum temperatures in dependence of the transmitted power showing that the power capability is even higher than 1 MW. In collaboration with the DeBeers Company, FZK is successfully performing metallization and brazing tests on cheap (gray) diamond samples with up to 100 mm diameter and 1 mm thickness (Fig. 6). Thermomechanic cycling at temperatures up to 450 °C did not lead to diffusion degradation of the Al-based brazing and no leaks occured. Finite element stress calculations including brazing/bonding stress show that the maximum principal stress is located at the window brazing (205 MPa) and is always present. During a 0.5 MPa overpressure event the maximum stress increases to 290 MPa and the transmission of 1 MW microwave power finally raises 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. Because the ultimate bending strength of CVD diamond is 600-900 MPa all stresses are well below the admissible limits.

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CVD DIAMOND FOR HIGH POWER GYROTRONS: CHARACTERISATION OF DIELECTRIC PROPERTIES

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1 Introduction

The development of the next generation of fusion devices (ITER, Wendelstein-7X) requires plasma heating systems (Electron Cyclotron Resonance Heating, ECRH) which provide mm-waves (110 - 180 GHz) in continuous wave operation at the 10 – 50 Megawatt level. Up to now the output windows of high power gyrotrons were the limiting component on the way to 1 MW source units. In the past and to the present different window designs have been developed using sapphire (cryogenically N_2 or Ne cooled), high resistivity grades of silicon (CO_2 gas pressure cooled) or boron nitride (water cooled, pulse operation). A promising alternative to these concepts is CVD diamond. The outstanding combination of low dielectric loss ($\tan\delta$) and of high resistance against thermal crack formation [1] as well as the possibility of a conventional water cooling at the edge of the window make CVD diamond to a persuasive solution.
Within the last year, the availability of high quality grades of CVD diamond has strongly increased. De Beers Industrial Diamond Division (Charters, Sunninghill, Ascot, England) and Fraunhofer Institut für Angewandte Festkörperphysik (Freiburg, Germany) demonstrated their capability in producing large sized free-standing CVD diamond wafers (up to 160 mm in diameter, more than 1 mm thick) [2,3]. In this work CVD diamond grades from Fraunhofer Institut (sample code IAF) and from De Beers (DB) were investigated for their dielectric properties. The $\tan\delta$ values achieved in the best grades were so low, that a new horizon for high power output for gyrotrons would open.

2 Experimental set-up

In the low frequency range (10^3 Hz up to 10^9 Hz) a LCR meter and a hybrid cavity were used to study CVD diamond discs (with a diameter up to 50 mm) for their dielectric parameters with a special focus on the temperature dependence of the loss tangent in the high temperature (300 K to 700 K) regime.
In the high frequency range (10^{10} Hz up to $2\cdot10^{11}$ Hz) two different Fabry-Perot resonator (open resonator) configurations were chosen. On the one hand at the Institute of Applied Physics (IAP), Nizhny Novgorod a (quasi-) spherical geometry allows the quantification of dielectric parameters by using the "length-tuning" method [4,5]. At Forschungszentrum Karlsruhe (FZK), Karlsruhe on the other hand resonators with a (quasi-) hemispherical geometry operate with the "frequency-tuning" method. In both cases the dielectric loss tangent is obtained by Q-factor measurements using Gaussian TEM_{ooq} modes.
A heating device between the two curved mirrors of the (quasi-) spherical Fabry Perot resonator at IAP offered the possibility of temperature dependent $\tan\delta$ measurements at about 150 GHz in the

high temperature (300 K to 700 K) regime. At FZK the temperature dependence of tanδ at 145 GHz in the low temperature (70 K to 370 K) regime was obtained and also the frequency dependence of tanδ with four individual (quasi-) hemispherical Fabry Perot resonators covering four different frequency ranges (10 – 20 GHz, 30 – 40 GHz, 90 – 100 GHz and 144 – 146 GHz) [6]. In addition the spatial homogeneity of large sized samples was characterised in a 30 – 40 GHz resonator and at 145 GHz in a specially focussed set up at FZK. For spatially varying losses, the apparent value arises from an integration of the loss over the disc plane weighted by the lateral beam profile and over the disc axis weighted by the field distribution in the material (contribution of the two faces).

3 Results

3.1 Low frequency range (10^3 Hz up to 10^9 Hz)

In the low frequency range it was not possible to determine the loss tangent at room temperature for high quality diamond wafers of both suppliers because the values were below the resolution limit of about 10^{-3}. At higher temperatures thermally exited processes led to an increase of tanδ above the resolution limit (**Fig. 1**). The activation temperature for these losses was shifted to higher values with increasing frequency.

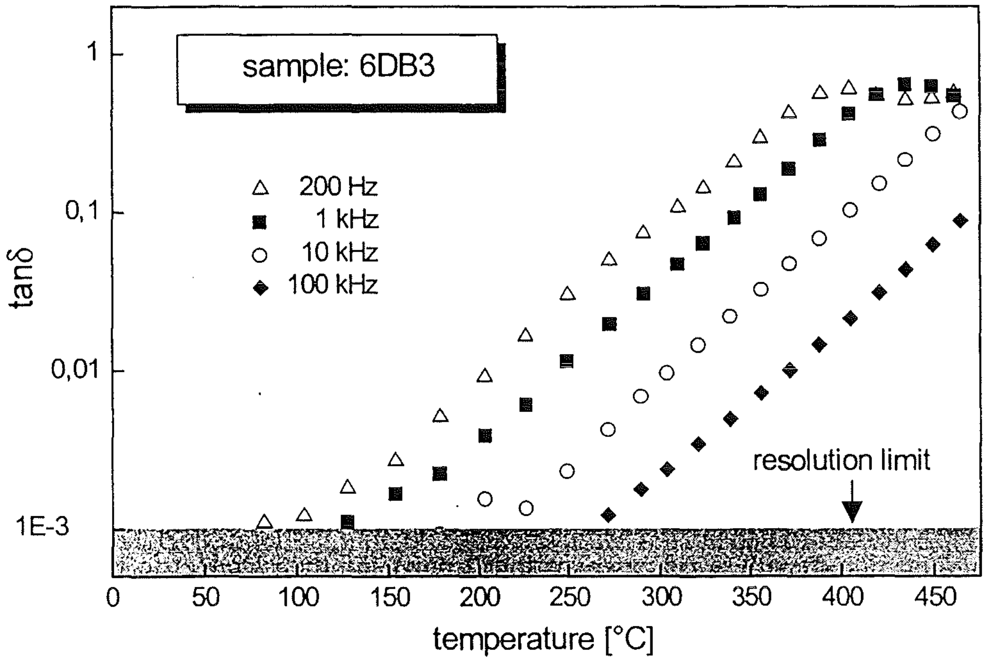


Fig. 1 Example for additional thermally exited losses.

3.2 High frequency range (10^{10} Hz up to 10^{11} Hz)

3.2.1 Temperature dependence

Between 70 K and 370 K all diamond qualities with loss tangent values varying from 10^{-3} to below 10^{-5} showed no significant thermally activated increase of loss tangent (**Fig. 2**). The reduction of tanδ of more than two orders represent also the progress in the growing process. The increase of scattering in loss tangent for the best qualities was due to the fact, that material was close to a perfect dielectric. For the 'Super-FZK' window (15DB1), the lowest loss tangent (up to now) for a CVD diamond material at 145 GHz (300 K) was obtained with $\tan\delta = 6\ (\pm 2)\cdot 10^{-6}$. In the whole low temperature regime the permittivity for all samples remained constant at $\varepsilon_r = 5.67\ (\pm 0.01)$.

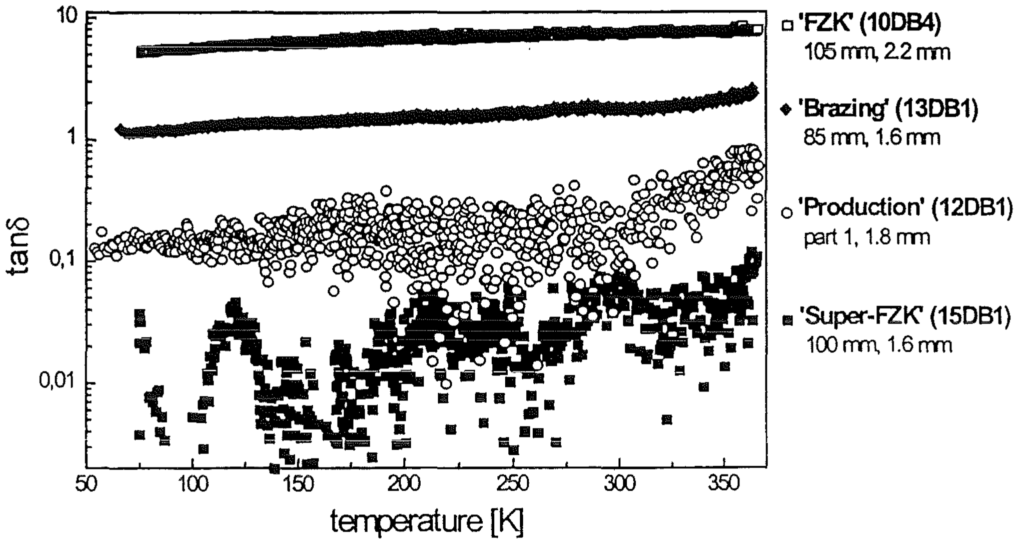
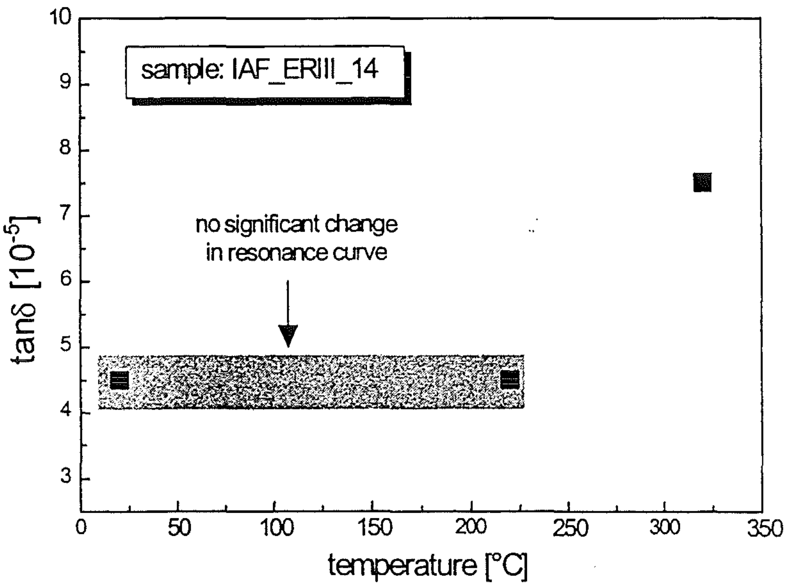


Fig. 2 Temperature dependence of CVD diamond windows from different grades (dimensions of disc given as diameter, thickness).

The measurement of tanδ in the high temperature regime on sample FHG_ERIII 14 [7] showed up to 225 °C no additional thermal activated losses, which goes along with results from Parshin et al [8]. For a temperature of about 300 °C - far above the application temperature for a high power



gyrotron window (below 100 °C in the centre of window) - an increased tanδ was observed.

Fig. 3 Temperature dependence of tanδ in CVD diamond at about 150 GHz.

3.2.2 Frequency dependence of loss tangent

To measure the frequency dependence of tanδ from 10 GHz to 145 GHz, it is mandatory to have large sized diamond wafer (> 70 mm in diameter) which are in addition lateral homogeneous in tanδ. The reason for this conditions comes from the increasing beam diameter at the plane mirror with decreasing frequency. For example if at lower frequencies outer disc sections with higher loss material are seen by the mm-wave (through the larger diameter of the beam), tanδ is shifted to a higher value.

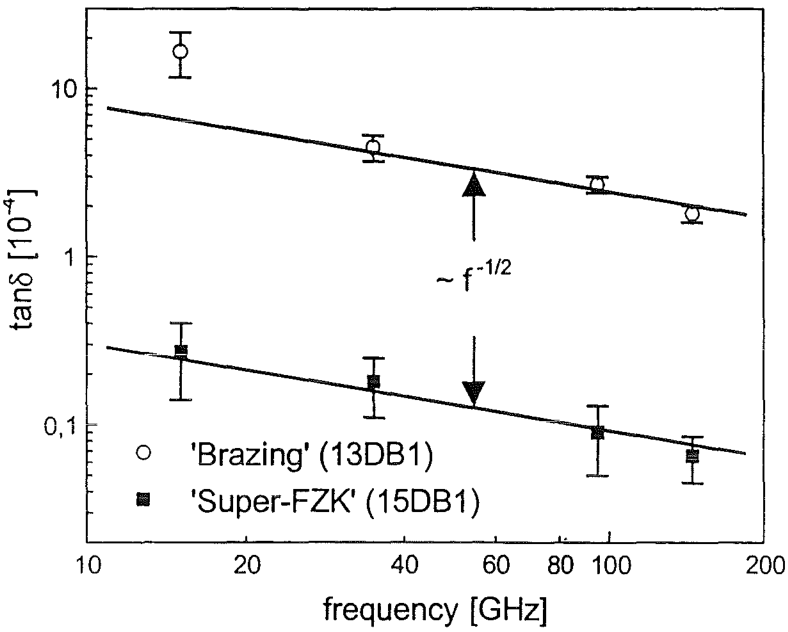


Fig. 4 Frequency dependence of the locally inhomogeneous 'Brazing' - window (13DB1) and the homogeneous 'Super-FZK' - window (15DB1).

The influence of such a local inhomogeneity (see also **Fig. 5**) in tanδ is shown in **Fig. 4** for the 'Brazing' window (13DB1). At 10 – 20 GHz there is a discrepancy from the empirical frequency law according to:

$$\tan\delta \propto \frac{1}{\sqrt{f}}$$

which was determined for the very homogeneous 'Super-FZK' window (see also **Fig. 6**).

3.2.3 Spatially resolved measurements

As it was shown in a previous paper [9,10], the nucleation and the growth face have different contributions to the loss, but bulk losses could superimpose these. For a gyrotron window all contributions to loss along the propagation direction of the travelling wave add, they are weighted by the lateral beam profile and lead to an effective loss value for the window. Therefore the homogeneity of the loss across the window is also crucial to avoid 'hot spots'.

An example for a CVD diamond with a 'hot spot' was the 'Brazing' window (**Fig. 5a**). For the area with the darker circles close to the centre a tanδ value which about twice above that one of the surrounding region was obtained. This could be seen also in the distribution diagram (**Fig. 5b**). The circles plotted in **Fig. 5a** do not represent the actual spot size of the measurement, but are demagnified for a clear lay-out. As an example for the really mapped area (circle diameter = real spot size ~ 6mm), **Fig. 6b** shows the measured losses in the 'Super-FZK' window (15DB1).

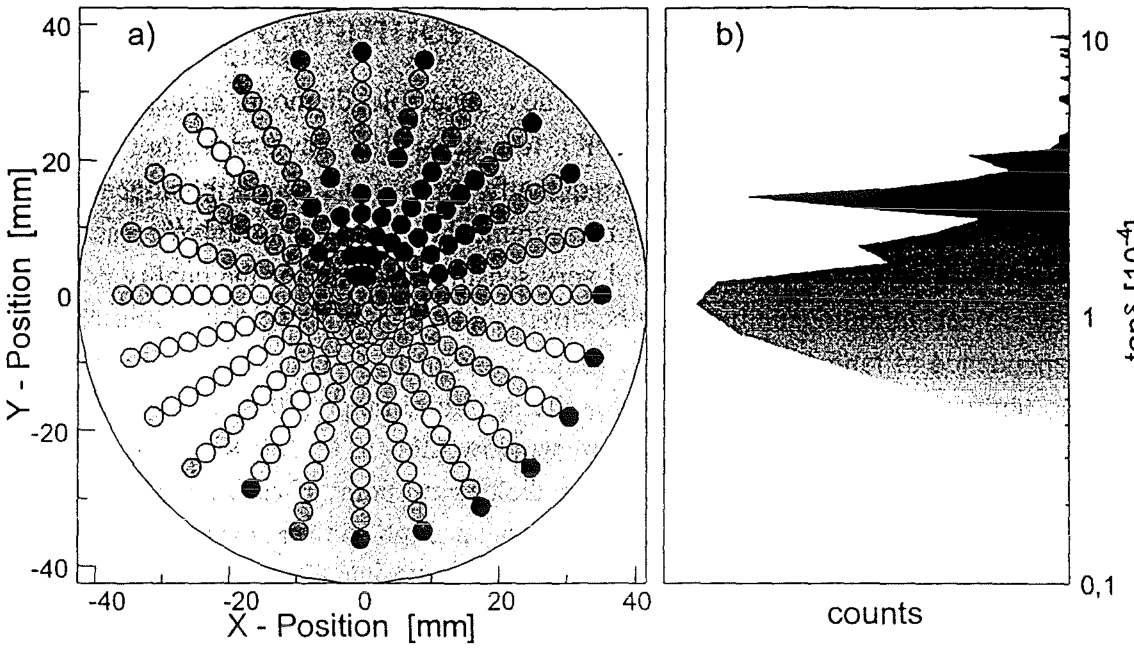


Fig. 5 **a)**'Brazing' window (13DB1) with a 'hot spot' and **b)** tanδ distribution.

The 'Super FZK' window (15DB1) was not only the CVD diamond with the lowest tanδ value, it is also very homogeneous in tanδ. In the central region (about 60mm in diameter), which is important for the application as a gyrotron window, tanδ values did not go beyond the 10^{-5} level (**Fig. 6a + c**).

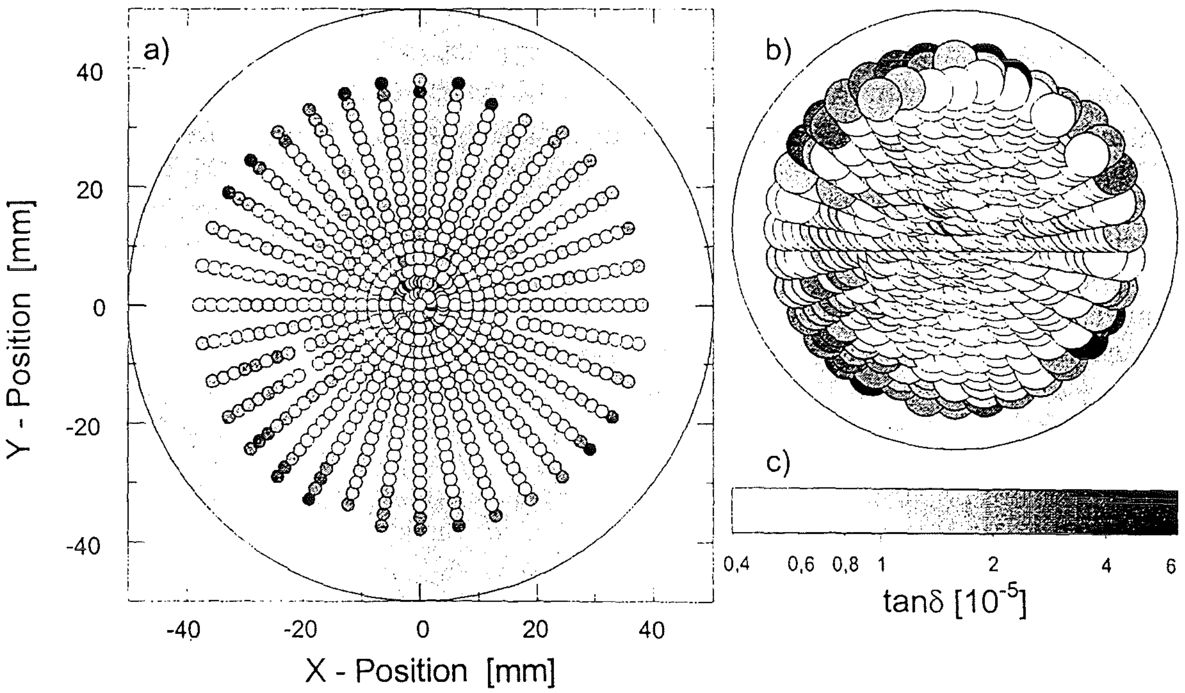


Fig. 6 **a)** Homogeneous 'Super-FZK' window (15DB1) with tanδ below 10^{-5} in the central region; **b)** mapped with real spot size of 6mm and **c)** tanδ grey scale.

4 Conclusions

Within this work it was demonstrated that the now available diamond grades from different producer reached very low tanδ values (at 300K): less than $5 \cdot 10^{-5}$ for grades from IAF and even below the 10^{-5} level for grades from DB. The quantification of the spatial homogeneity of large sized diamond windows by position resolved measurements of tanδ is not only necessary to avoid 'hot spots' in a window, it is also a useful and very sensitive method for the quality control of the CVD process. Local inhomogeneities in the loss could superimpose the empirical obtained frequency dependence of tanδ ($\sim f^{-1/2}$) in the range between 10 GHz and 145 GHz. Tanδ showed practically no temperature dependence in a broad temperature range (70 K up to 500 K), only far above the application temperature as a gyrotron window (300 K to 350 K) additional thermally activated losses were observed. Therefore from the dielectrical point of view a window design with CVD diamond is no longer the limiting issue for a high power gyrotron (1 MW, CW, at 110 - 180 GHz). With the best grades (homogeneous 'Super-FZK' window from DB with $\tan\delta = 6\ (\pm 2) \cdot 10^{-6}$) even an output power level for gyrotrons of about 2 MW would be possible.

5 Acknowledgement

This work has been performed in the framework of the Nuclear Fusion Project of the Forschungszentrum Karlsruhe within the European Fusion Technology Programme. Joint measurements were promoted by the bilateral scientific cooperation programme of the German and Russian government (WTZ # RUS-519). The authors would like to thank P. Koidl and C. Wild from Fraunhofer Institut für Angewandte Festkörperphysik (Freiburg, Germany) and also R.S. Sussman and J.R. Brandon from De Beers Industrial Diamond Division (Charters, Sunninghill, Ascot, England) for supplying of CVD diamond samples and for helpful discussions.

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CVD DIAMOND WINDOWS FOR HIGH POWER GYROTRONS

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Abstract

The recent development of large size CVD diamond wafers with low mm-wave losses has enabled the manufacture of output windows for Megawatt gyrotrons. The progress in the window development is illustrated with a 100 mm dia. x 1.6 mm thick window disc mounted on double metal cuffs with a UHV compatible diffusion bond. In the disc, excellent properties ($\tan\delta < 10^{-5}$, good homogeneity) were established. Further up-scaling potential is demonstrated with a 120 mm dia. x 2.2 mm CVD diamond window disc.

Introduction

The challenge of realising output windows for high power gyrotrons that generate in continuous wave (CW) operation power levels of at least 1 MW has called for extensive materials development and window design activities in the recent years. In the search to overcome the limitations for transmissible mm-wave power for Electron Cyclotron (EC) wave systems, which are projected to serve in the next step fusion machines at frequencies between 90 GHz and 170 GHz, a number of different window designs have been developed based on sapphire (cryogenically N_2 or Ne cooled), high resistivity grades of silicon (CO_2 gas pressure cooled) or boron nitride (water cooled, restricted to EC wave pulses).
As excellent resistance against thermal crack formation is one of the major materials performance criteria [1], diamond received particular attention when the possibility of manufacture of large size diamond plates by the Chemical Vapour Deposition (CVD) process was demonstrated. Apart from the outstanding thermal and mechanical parameters, low levels of mm-wave absorption which is parameterised by the dielectric loss tangent ($\tan\delta$) must be attained. The first experience with early CVD diamond material, often 'black grades', was rather discouraging, as $\tan\delta$ levels were prohibitively high ranging between 10^{-3} and 10^{-2} in the interesting frequency range [2,3]. Progress in material development became substantial, after special 'white' grades were identified which showed $\tan\delta$ levels below 10^{-4} [3,4] and thus became competitive to Sapphire. When the first full size windows were produced successfully with losses in the range of 10^{-4}, it became evident that inhomogeneities in mm-wave absorption along the disc axis, as well as in the radial direction, were of concern [5]. In the present paper, window material from further refined CVD diamond grades is presented which combine good homogeneity with very low $\tan\delta$ level and which can be mounted into window structures which opens a new horizon for high power gyrotrons.

Characteristics of the window material

The new types of CVD diamond material were studied in form of two large discs. The first one was produced to serve as 'standard window type' with 4 inch diameter with actual geometries of 100 mm x 1.6 mm. It was given the internal name 'Super-FZK', in continuation of a series of names starting with the 'FZK' disc presented previously [5]. The second one was produced to investigate and to demonstrate the growth process adopted for even more extended window geometries, such as for Brewster windows. This disc was denominated 'Star of FZK', its dimensions are 120 mm x 2.2 mm. The older 'FZK' disc which will serve as a reference component in this paper had the size of 105 mm x 2.2 mm.
All three discs were produced using the microwave plasma assisted CVD (MPACVD) technique [6]. Reactor geometry and processes were developed specially for the synthesis of large diameter, high quality diamond discs. Following synthesis, material was removed from both growth and nucleation surfaces by lapping to achieve a thickness tolerance better than ± 20 µm and surface Ra values less than 200 nm.

Dielectric measurements

For the measurements of mm-wave properties, two different set-ups were used which determined the permittivity (ε_r) and the dielectric loss tangent at 145 GHz based on the open resonator technique with a quasi-hemispherical resonator geometry. The temperature dependent data were obtained with a fixed mirror system installed in a liquid flow cryostat, spatially resolved measurements (line scans, mapping) were obtained with a new system where the specimen can be shifted together with the plane mirror over the mm-wave beam by computer driven positioning devices. Details on both measuring facilities and their resolution has been published recently elsewhere [7].

The temperature dependent measurements between 70 K and 370 K showed for all three discs no significant changes with temperature. Whereas no difference occurred in the permittivity, which amounted to ε_r = 5.67 (± 0.01), striking reductions in tanδ of more than one order of magnitude could be observed for the two new discs. The increased scattering in loss tangent data for the 'Super-FZK' window disc was due to the fact, that material came close to a perfect ultralow loss dielectric. Here the lowest loss tangent so far reported for a CVD diamond material at 145 GHz (300 K) was obtained with tanδ = 6 (±2)·10^{-6}. Even in the large 'Star of FZK' losses hardly exceeded 2 · 10^{-5}.

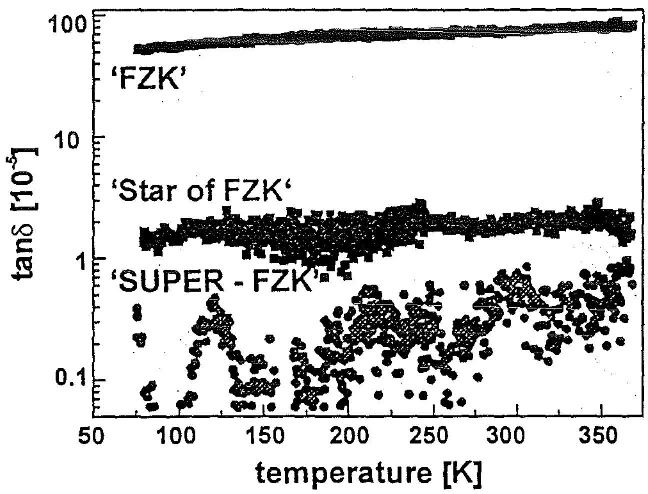

Fig. 1 Temperature dependence of tanδ at 145 GHz for the 'FZK', 'Star of FZK' and 'Super-FZK' window discs.

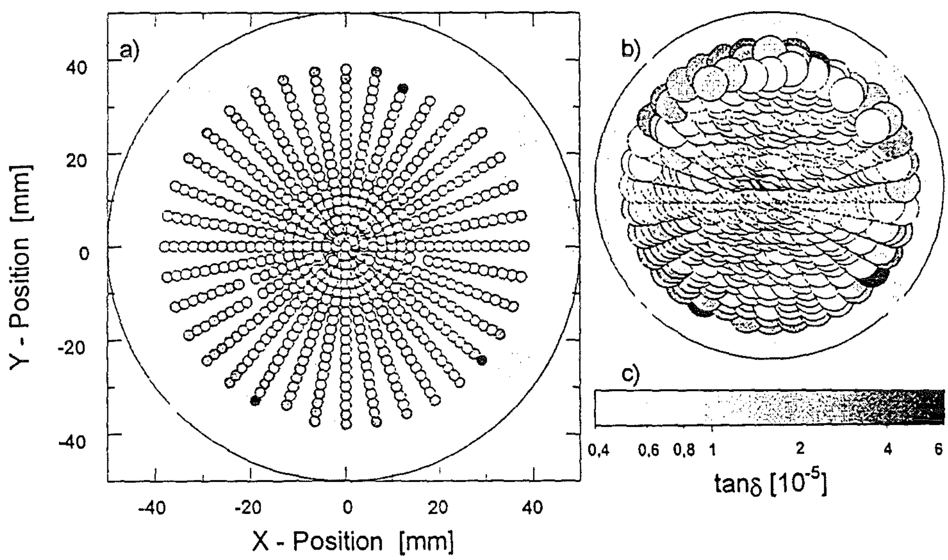

Fig.2 Tanδ mapping for the 'Super-FZK' window disc: a) demagnified spot size, b) actual spot size of 6mm and c) tanδ grey scale.

It was clear from the earlier paper [5] that different surface contributions to the loss typically arise at the nucleation and the growth face, which may be eliminated by polishing off an appropriate material quantity, but bulk losses could superimpose these. In the present new discs, no significant differences were observed along the growth axis by alternative placing the nucleation and the growth face into the resonator.

As for the distribution of the losses observed across the area of the disc, the tanδ map plotted in Fig. 2 shows the good homogeneity of losses in the 'Super-FZK' window disc. The circles plotted in Fig. 2a do not represent the actual spot size of the measurement, but are demagnified for a clear lay-out, whereas for the actually mapped area (circle diameter = real spot size ~ 6 mm) is given in Fig. 2b. Obviously in the central region (about 60 mm in diameter), which is the most critical for a gyrotron window, tanδ values did not go beyond the 10^{-5} level. A line scan across the 'Star of FZK' window disc gives evidence of constant low loss area ($\tan\delta < 3 \cdot 10^{-5}$) in the inner area of at least 80 mm dia (cf. Fig.3). The increase in the outer area may be enlarged by diffraction at the disc edges.

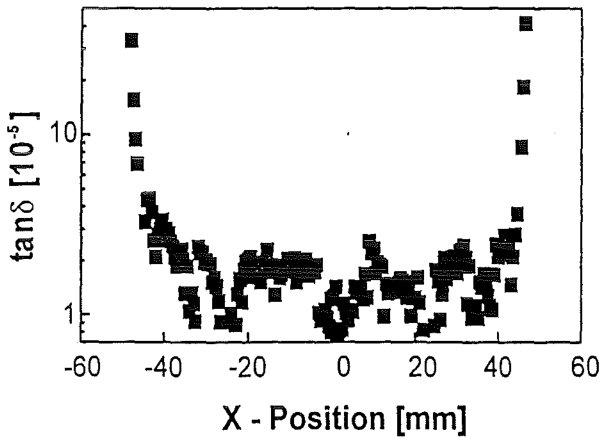

Fig.3 Line scan across the diameter of the 'Star of FZK' window disc.

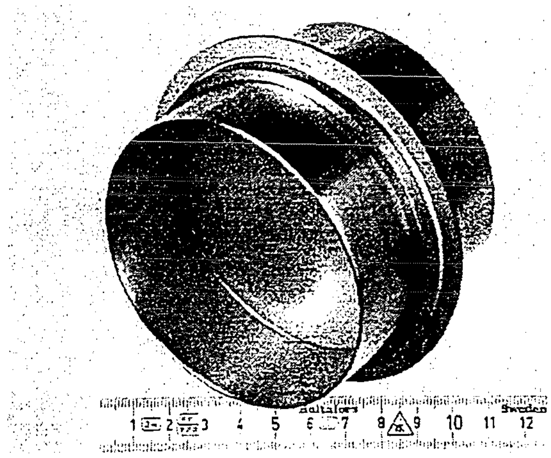

Fig.4 The 'Super-FZK' disc mounted into a test structure for high power mm-wave tests.

Mounting of the window material

To prepare for high power testing, the 'Super-FZK' window disc was mounted on double inconel cuffs using an ultrahigh vacuum (UHV) compatible, aluminium based diffusion bond. The leak rate for the assembly was measured at $< 10^{-9}$ mbar·l/s. The cuff dimensions were chosen to allow a clear aperture of transmission of 80 mm in diameter and sufficient material at the edge for efficient water cooling. The whole cuff assembly was then baked at temperatures up to 450°C for more than 24 hours, which represents a typical out-gassing cycle, and subsequent tests confirm vacuum tightness.

Acknowledgement

Parts of this work have been performed in the framework of the Nuclear Fusion Project of the Forschungszentrum Karlsruhe within the European Fusion Technology Programme.

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DIELECTRIC PROPERTIES OF CVD DIAMOND FOR RADIOFREQUENCY WINDOW APPLICATIONS

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Free-standing discs of CVD diamond were produced with a new technique by which windows with a diameter up to 6 inches can be formed. Specimen sets grown with this technique were studied for their dielectric parameters in a wide frequency range from 10^3 Hz up to 10^{11} Hz by different methods (LCR meter, Hybrid cavity, Fabry-Perot resonator) at room temperature. In addition the temperature dependence of loss tangent (tanδ) was investigated in the cryogenic (70 K to 300 K) and high temperature (300 K to 700 K) regime.
In the best grades, values of tanδ lower than $5 \cdot 10^{-5}$ at 145 GHz (300 K) were obtained.

1. INTRODUCTION

Parallel to other high performance windows developed, e.g. for high power CO_2 lasers, there are intensified efforts to establish CVD diamond as an advanced window material for high power microwave tubes, e.g. gyrotrons[1]. For gyrotron output windows various concepts based on sapphire (cryogenically N_2 or Ne cooled), high resistivity grades of silicon (CO_2 gas pressure cooled) or boron nitride (water cooled, pulse operation) were followed. Up to the present these windows form a limiting component for the realisation of gyrotrons providing 1MW continuous wave power at 110 GHz – 180 GHz. Such units are needed for plasma heating systems (Electron Cyclotron Resonance Heating, ECRH) in the next generation of fusion devices (ITER, Wendelstein-7X) where a total mm- wave power between 10 – 50 Megawatt has to be provided by a manageable number of gyrotrons.

The outstanding combination of low microwave absorption[2], high thermal conductivity[3], high resistance against thermal crack formation[4,5,6] and consequently the perspective of a conventional water cooled window promise CVD diamond to become the ultimate window solution not only for high power gyrotrons but also for other high power tubes such as klystrons. A particular aspect of radio frequency window application are the demands for large diameter (up to 4 or 5 inches) and thickness (values 1 - 2 mm).
In this work it will be demonstrated that a novel CVD technique is available to produce large CVD diamond discs. Discs grown with this technique show very low mm-wave absorption documented by loss tangent values lower than $5 \cdot 10^{-5}$ at 145 GHz and room temperature.

2. SAMPLE PREPERATION

The free-standing diamond discs under investigation have been prepared using microwave-plasma-assisted chemical vapour deposition (CVD). The deposition was carried out in a novel microwave plasma system which uses an ellipsoidal cavity to generate very intense, spatially extended plasmas[7]. The performance of these microwave reactors have been optimized with respect to long term stability and homogeneous growth on large areas.

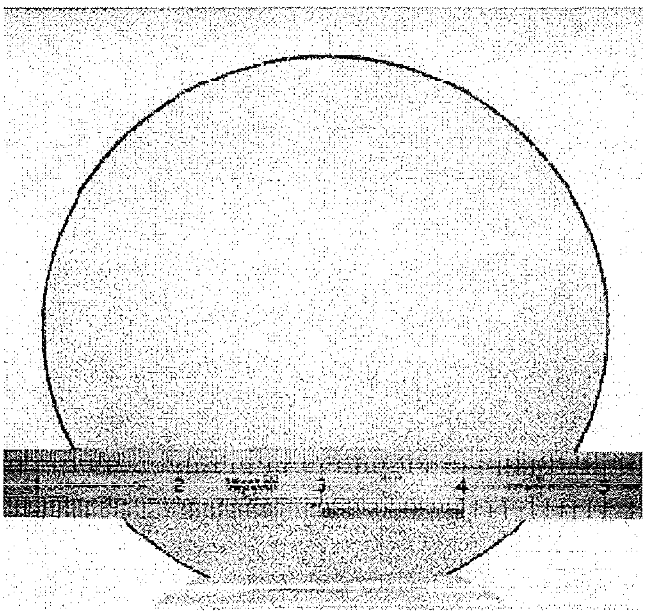

FIGURE 1

White 4" diam. CVD diamond window.

Two versions of this CVD system have been employed for the preparation of diamond discs: in a 2.45 GHz system powered with 6 kW microwave radiation discs with 2" and 3" in diameter are prepared, whereas in a 915 MHz system equipped with a 60 kW microwave generator diamond discs with up to 6" in diameter are produced. FIGURE 1 shows an unpolished free-standing high-grade diamond disc with 4 inch diameter and 1 mm thickness. Corresponding discs with thickness up to 1.8 mm have been processed.

After deposition the diamond discs are separated from the substrate and laser-cut to the desired shapes and dimensions using a Q-switched Nd:YAG laser. Since the surface of as-grown diamond films is very rough, grinding and polishing techniques are applied to obtain a smooth surface finish and a homogeneous thickness distribution.

By using purified gases and optimising the process conditions, the quality and phase-purity of the CVD diamond discs have been considerably improved. Samples prepared under optimum growth conditions exhibit properties approaching those of perfect type IIa diamond crystals. This includes an excellent broad-band optical transparency and a thermal conductivity exceeding 20 $Wcm^{-1}K^{-1}$ at room temperature[3].

3. EXPERIMENTAL SET-UP

In the lower radiofrequency range (10^3 Hz up to 10^9 Hz) a LCR meter and a hybrid cavity were used to study the dielectric properties (permittivity ε_r and loss tangent $\tan\delta$) of CVD diamond discs (with a diameter up to 50 mm) The studies were motivated to establish the typical temperature dependence of $\tan\delta$ in the high temperature (300 K to 700 K) regime.

In the mm-wave range, ε_r and $\tan\delta$ are assessed by two (quasi-) hemispherical Fabry Perot resonators (30 – 40 GHz, 144 – 146 GHz) analysed by the "frequency-tuning" method[8]. The dielectric parameters are obtained by Q-factor measurements using Gaussian TEM_{ooq} modes. In addition the temperature dependence of ε_r and $\tan\delta$ was tested at 145 GHz studying the low temperature (70 K to 340 K) regime.

4. EXPERIMENTAL RESULTS

4.1. Lower radiofrequency range

For high quality diamond wafers it was not possible to determine the dielectric loss tangent at room temperature, because the tanδ values were below the resolution limit of about 10^{-3}. At higher temperatures thermally activated processes led to an increase in the absorption and substantial tanδ values could be assessed (FIGURE 2). The efficiency of the thermal activation of increased losses shifts to higher temperatures with increasing frequency.

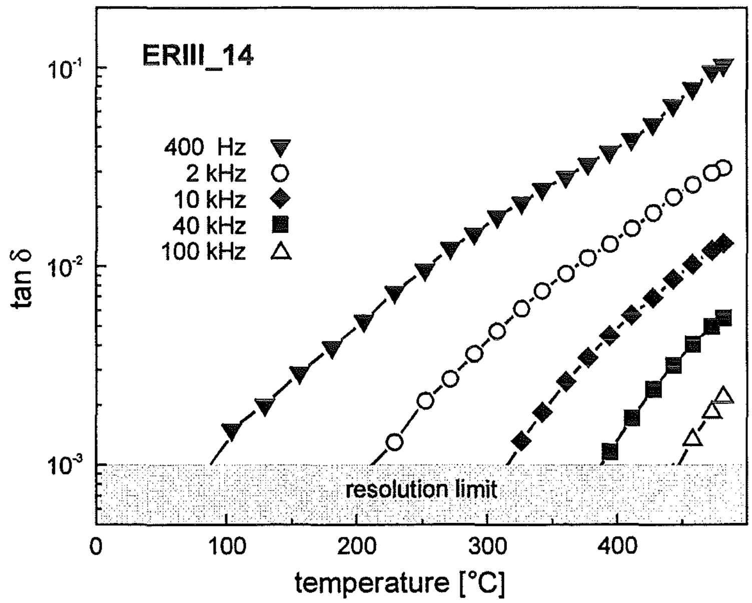

FIGURE 2
Example for additional thermally activated radio frequency losses in CVD diamond.

4.2. Millimeter-wave range

At room temperature diamond wafers from different grades were tested for their dielectric loss. The test samples of 2" diameter were polished on growth surface and the measured loss values ranged from $2 \cdot 10^{-4}$ to $2 \cdot 10^{-2}$ at 30 - 40 GHz and from less than $5 \cdot 10^{-5}$ up to $1 \cdot 10^{-3}$ at 145 GHz (TABLE 1).

wafer	ERIII_14	ERIII_20	ERIII_49	ERVI_19
30 – 40 GHz	2	35	-	-
145 GHz	0.4	3	5	< 0.5

TABLE 1
Loss tangent values [10^{-4}] for different CVD diamond grades.

The permittivity was assessed to be 5.67 (±0.01) for all measurements at room temperature. Over the whole low temperature regime, this permittivity value was maintained as shown in FIGURE 3.

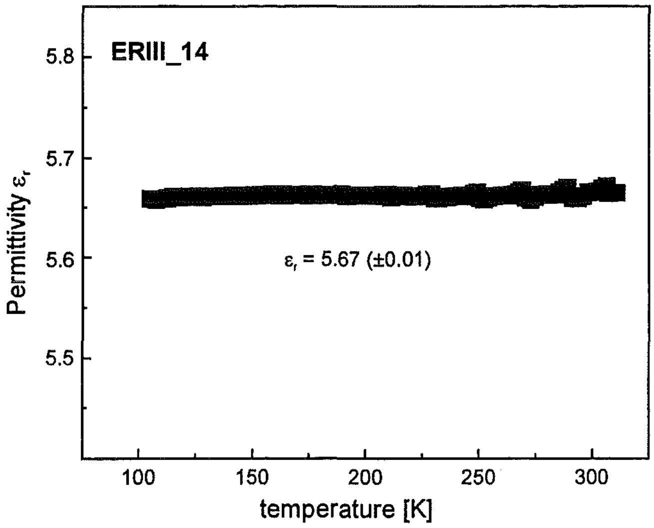

FIGURE 3
Permittivity in the low temperature regime.

A particular aspect is the homogeneity of the dielectric loss, which is not always given in CVD diamond[2,9]. Therefore the measured tanδ value comes from an integration of the loss over the disc plane weighted with the lateral beam profile and over the disc axis weighted with the field distribution in the material. Because of standing wave pattern within the thickness of the diamond sample different contributions from the growth and nucleation side can be observed in the apparent loss as shown for disc ERIII_14

(FIGURE 4). As a consequence, in this particular disc a reduction of the loss level can be expected when removing high loss material from the 'as grown' nucleation side.

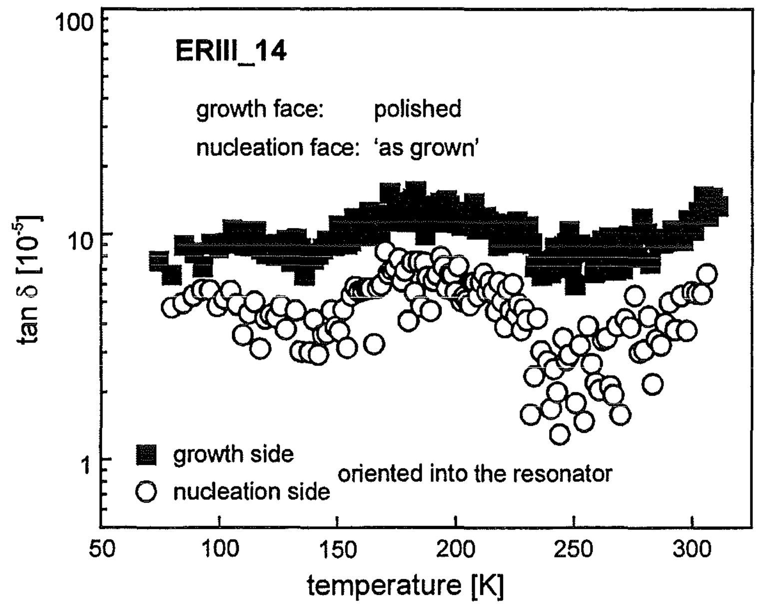

FIGURE 4
Orientation dependent dielectric loss tangent in low temperature range.

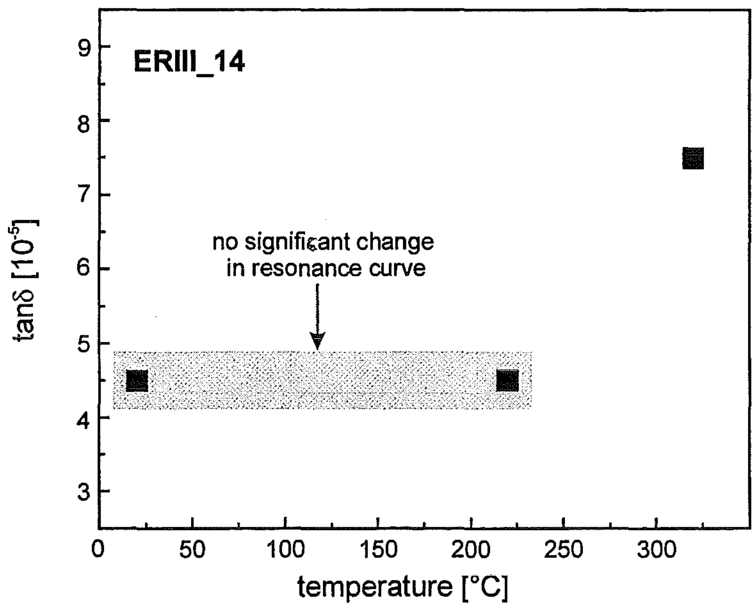

FIGURE 5
Temperature dependence of tand in the high temperature regime at about 150 GHz.

The measurement of tanδ in the high temperature regime did not give any evidence for additional thermally activated losses up to 225°C, which goes along with previous results obtained for different grades[10]. Only for a temperature of about 300°C – which is well above the application temperature for a high power gyrotron window (below 100°C in the centre of window) - an increase in tanδ was established.

5. CONCLUSIONS

A new technique, which is able to produce large sized CVD diamond wafers with a diameter up to 6 inches and with thickness up to 1.6 mm was used to fabricate test disc for high power radio frequency window applications. In the best tested discs low dielectric losses were found to be kept at a tanδ level below $5 \cdot 10^{-5}$ at 145 GHz (300 K). Taking into account that a further reduction of this value is possible by removing high loss material from the nucleation side this technique has the potential to provide discs which are comparable to the best grades of CVD diamond measured up to now ($6\ (\pm 2) \cdot 10^{-6}$, 145 GHz, 300 K)[2]. In conclusion the requirement for an advanced window material operating in Megawatt gyrotrons is adequately met from dielectric point of view.

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MECHANICAL PROPERTIES OF FREE-STANDING CVD DIAMOND WAFER

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Free standing CVD Diamond wafers (50 mm diameter) of various thickness were grown by Microwave Plasma Assisted Chemical Vapour Deposition (MWPACVD). From these wafers of different quality ('white grade' material for optical and radio frequency applications and 'black grade' material for thermal management applications) as defined by colour, Raman spectroscopy and thermal conductivity discs were laser cut and studied for their fracture strength using the ball on ring test. Compared to standard technical ceramics (such as alumina), CVD diamond of a given quality has a high median bending strength and a narrow distribution of the strength values (very high Weibull modulus m > 20). Yet both grades exhibit a reduction in strength with increasing thickness (always growth side under tension during the test): 'white grade' material (about 100 µm to 500 µm thick) ranging from about 850 MPa to 550 MPa and 'black grade' material (100 µm to 400 µm thick) ranging from 1400 MPa to 1000 MPa. This dependence is apparently caused by a gradient in the microstructure along the growth direction. For both qualities the strength was found to be much higher when nucleation is oriented to the ring (nucleation side under tension), where values as high as 3500 MPa occur in 'black grade'. This can be understood in terms of macroscopic deformation caused by high internal stresses. Also, it was found that the surface finish achieved by lapping and polishing procedures produced no major influence on the strength level.

1. INTRODUCTION

The growing interest in CVD diamond as an advanced window material ranging from optical windows (e.g. for high power CO_2 couplers[1]) to microwave frequency windows (for high power gyrotrons or klystrons) is motivated by a combination of low absorption[2] and high thermal conductivity. The application of bulk CVD diamond in the field of thermal management where diamond is already established for mounting high power laser diodes is also an expanding market, one example being the development of diamond support rods for travelling wave tubes. For such applications thermal stresses caused by temperature gradients and additional mechanical loads are obviously of primary concern.

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Thermal stresses in window applications are related to temperature gradients arising from locally varying power loads due to the field distribution in the beam and due to the spatial inhomogeneity of the window material. Furthermore pressure gradients across the window can form additional stresses. If the sum of these stresses reaches the fracture strength level of the material, spontaneous failure will occur. Other failure modes are subcritical crack growth under static load and cyclic fatigue but these are out of the scope of this work. At present only a few data for CVD diamond concerning these mechanical properties are available[3,4,5]. In this paper, strength data of CVD diamond determined by the biaxial ball on ring test as a function of diamond quality, surface finish and thickness are presented. Special emphasis is laid on the anisotropy of the microstructure (as a result of columnar grain growth) on the observed strength properties.

2. DIAMOND PROCESSING AND GROWTH

2.1. Diamond Growth

CVD diamond manufactured at GMMT is grown by the Microwave Plasma Assisted Chemical Vapour Deposition method. The first stage of this process is the nucleation of the substrate material, usually but not exclusively silicon. The substrate is given a polish using fine diamond grit. The substrate is then placed in a chamber, which is evacuated. Methane heavily diluted in hydrogen is then fed into the chamber to a pressure of around 100 Torr. A plasma is then struck by a 5 kW microwave discharge (operating at 2.45 GHz). This discharge forms both the hydrocarbon radicals, which are the precursors to diamond growth and atomic hydrogen which promotes the formation of diamond. Small amounts of oxygen may also added to the feed gas. If the methane fraction is increased the growth rate increases, but in turn the diamond quality is reduced. The optical grade CVD diamond "white diamond" is grown with a lower methane fraction than the mechanical grade diamond "black diamond". It must however be emphasised that these diamond growth recipes represent just two points in a continuum of possible diamond growth recipes. The growth itself can be split into two stages: nucleation and growth proper. Before a continuous film is produced, which then grows in a columnar fashion, the individual diamond seed crystals have to nucleate and coalesce to form a continuous film. This is the nucleation stage, where growth is radially outwards from the seed crystals. The diamond on the nucleation side of the diamond wafer (the first material to grow) is fine grain, and therefore more disordered crystalline material. Many applications require the removal of this material, which is done by polishing. The growth side of the diamond wafer is defined as the opposite side to the nucleation side and generally consists of larger grains that have grown out at the expense of neighbouring grains.

2.2. Diamond quality

It is well known that CVD diamond can be grown to various qualities. In general the quality of a CVD diamond sample is a function of the amount of non-diamond carbon it contains (graphitic, amorphous carbon etc.), extrinsic impurities like Nitrogen and Silicon and to some extent the average grain size (number of grain boundaries). Gaseous impurities such as nitrogen can generally be controlled to very low levels (ppb) by gas purification and good quality vacuum equipment, Si and other elements may be etched from the reactor furniture, but in general with careful reactor design the plasma can be kept remote from most surfaces and in this way the extraneous impurities kept extremely low. Grain size is to a large extent a function of the diamond thickness and is therefore a variable in CVD diamond that cannot easily be controlled directly. Non-diamond carbon is probably the most important impurity to control because diamond is not the intrinsically stable form of carbon at the temperatures and pressures at which growth occurs. One therefore has to produce a growth environment in which all forms of carbon bonding except diamond have difficulty surviving. This is essentially achieved by creating a hydrogen plasma with a high concentration of atomic hydrogen which tends to favour the formation of diamond over other forms of carbon. Experience shows that a lot about the quality of diamond can be assessed form it visible properties. It is beyond the scope of this paper to describe in detail the various characterisation techniques that can be applied to diamond although a good reference can be found in Ref. 6. For the purposes of this work two grades of diamond were chosen for test which were easily distinguished on the basis of their colour. White diamond is a visibly transparent grade of diamond grown in the CVD equipment described above at a growth rate between 2 to 4 μmh^{-1}(at 800 - 900°C), whereas black diamond is visibly opaque (or slightly translucent) having a black appearance and grown at a much faster rate (10 to 14 μmh^{-1} at 800 - 900°C). Generally speaking one can say that 'black' diamond contains more non-diamond carbon than 'white'. It is also true to say there is a continuum of quality between these two extremes and also some variation within diamond that is nominally 'white' or nominally 'black'. However in terms of mechanical properties the assumption was made that 'white' and 'black' diamond represented two ends of the spectrum of quality and therefore their mechanical properties would be a good indication of the differences that exist at the two ends of the purity spectrum. Typical thermal conductivity values observed for the two extremes are of 1000 - 1500 $Wm^{-1}K^{-1}$ for 'black' and 1800 - 2100 $Wm^{-1}K^{-1}$ for 'white' diamond; Raman trace characteristic for both grades are shown in FIGURE 1 and FIGURE 2. All the 'white' or 'black' samples grown for these trials were grown under essentially identical conditions the only variable being their thickness which was simply a function of the growth time.

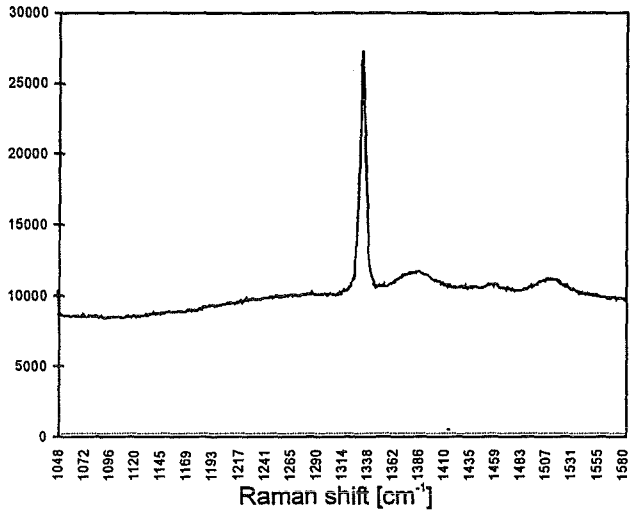

FIGURE 1
Raman trace of 'black' diamond.

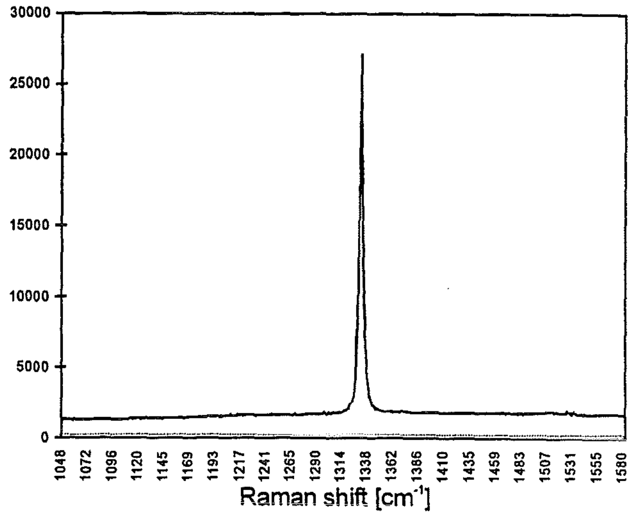

FIGURE 2
Raman trace of 'white' diamond.

2.3. Diamond Processing

Laser cutting was used to produce 11 mm diameter discs from the as grown 50 mm diameter wafers. A Nd:YAG lasers was used to cut CVD diamond. The diamond exposed to the laser beam is graphitized and vaporised due to the intense heat. Most of the carbon is converted to CO_2 as the cutting is carried out in air. Some of the diamond samples tested were either lapped or polished. It is often the case that the growth surface is too rough to be used for commercial applications. The production of a smooth flat surface is carried out using a mechanical process in two distinct stages: lapping and polishing. Lapping is the stage concerned with producing a globally flat surface, but one which still contains many digs and crevices within the surface. Polishing is concerned with producing a specular finish. Lapping and polishing is generally achieved with different grades of diamond powder using proprietary techniques. The roughness average after lapping is about 100 nm, whereas after polishing it will be better than 40 nm.

2. BALL ON RING TEST

The mechanical strength of free standing CVD diamond discs has been determined by using the ball on ring method[7]. Especially important for diamond is the fact that the edge quality has negligible influence on the stress distribution within the support ring to avoid time and cost intensive sample preparation steps. The diamond disc (about r_{disc} = 5.5 mm) is centred on the support ring (r_{ring} = 5 mm) with careful attention paid to whether the growth or nucleation side of the disc is oriented to the ring, because this side is under tension. During the test an increasing load is transmitted to the disc via a ball (r_{ball} = 5 mm) up to spontaneous failure of the sample. The loading rate chosen was very high (up to 500 $MPas^{-1}$) to avoid subcritical crack growth. Assuming a Hertzian contact between the ball and the diamond disc, the fracture strength could be determined from the maximum outer fibre stress at failure, which arises at the centre of the bottom surface as suggested by Ref. 8. The thickness of the discs were measured from SEM pictures of the fracture cross sections close to the centre. For 'as grown' specimens the median distance between the flat nucleation side and the valleys of the growth side were used for the calculation. The values for Young's modulus (1050 MPa) and Poisson's ratio (0.1) which are required for strength evaluation were taken from literature.

3. EXPERIMENTAL RESULTS

3.1. Fracture mode

The fracture patterns shown in FIGURE 3 are typical for both grades (growth face towards ring), demonstrating crack propagation initiated at the centre, which allows an accurate determination of the fracture strength taking the maximum fibre stress.

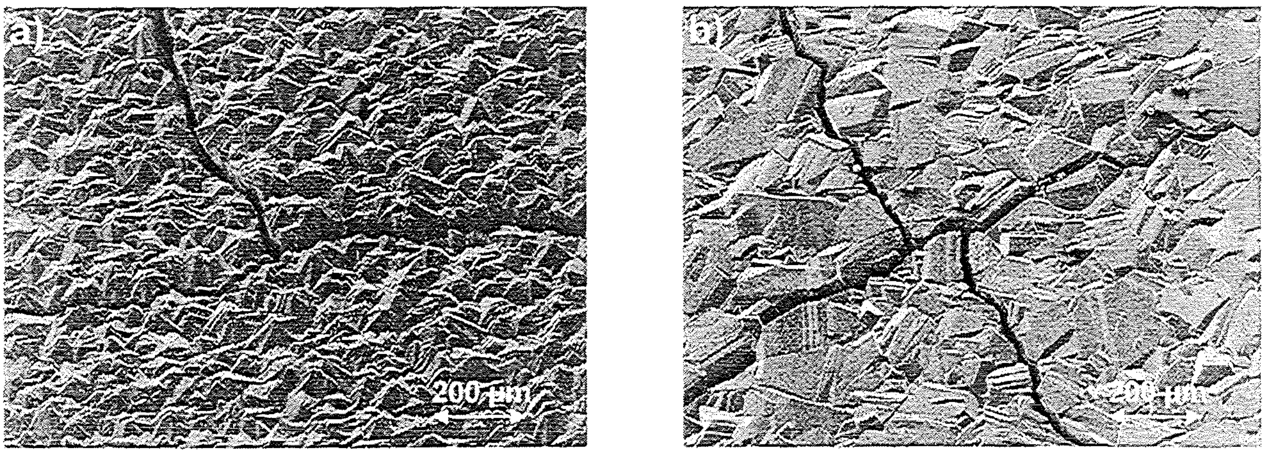

FIGURE 3
Fracture patterns of a) 'black grade' (disc from b16) and b) 'white grade' (disc from w11) CVD diamond (growth side). Discs were tested with growth side oriented towards the support ring.

Typically three or four cracks run from the centre to the edges. The fracture path is a mixture of intercrystalline and transcrystalline crack propagation[9]. For 'white grade' material, especially in the highly stressed centre, predominantly intercrystalline crack propagation was observed. For 'black grade' material an enhanced tendency to transcrystalline crack propagation was found. In both grades the median crystallite size increase with specimen thickness characteristic for their columnar grain growth.

4.2. Strength

The influence of the surface finish on the strength was tested by manufacturing wafers with different surface finish: ′as grown′ (wafers b19/b20/b21), lapped (b02) and polished (b04). The results (median strength value of about 5-7 discs for each wafer) do not give evidence of any major variation with the surface quality: median fracture strength for b19/b20/b21: 1050 ± 50 MPa, for b02: 980 ± 70 and for b04: 990 ± 30 MPa. No apparent reduction of the strength level induced by the surface machining was detected. Strength measurements on discs of both grades varying in thickness between 100 μm and 500 μm were performed. The comparison between both qualities for growth side oriented to the ring is shown in FIGURE 4 using about 8 discs from each wafer. Only one or two were used for the tests with nucleation side under tension presented in FIGURE 5. The strength values of 'white grade' material are significantly below the values of 'black grade'. For both grades with growth face supported, a decrease in strength with increasing thickness was observed, mainly influenced by the gradient in microstructure (columnar grain growth) parallel to growth direction.

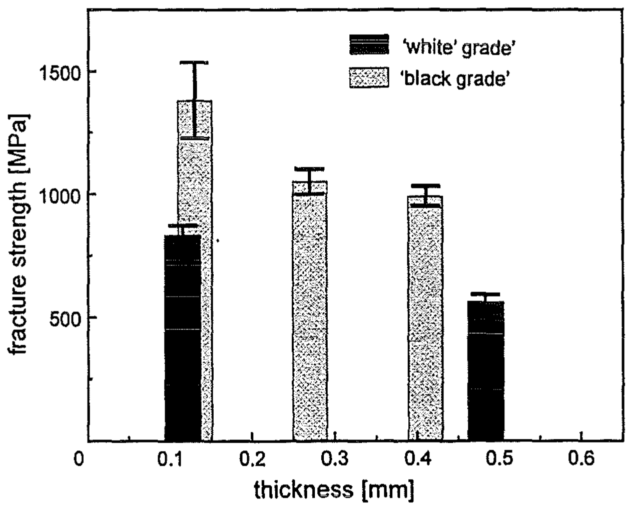

FIGURE 4
Influence of thickness: growth side oriented towards the support ring.

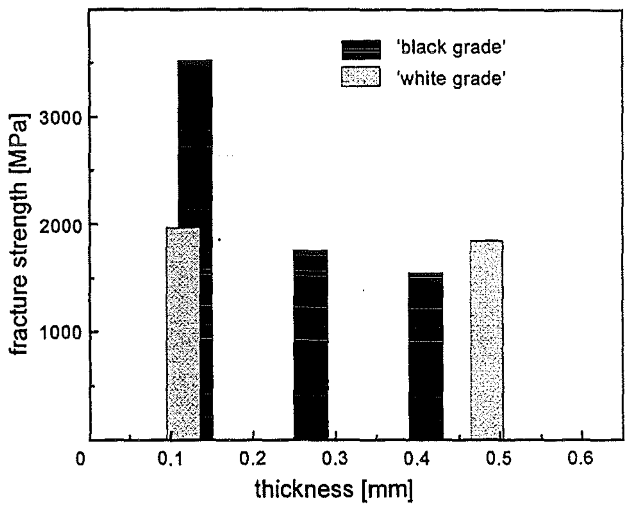

FIGURE 5
Influence of thickness: nucleation side oriented towards the support ring.

For the nucleation side in tension the two grades show higher strength values than for the other orientation. The thickness dependence of strength shown in FIGURE 5 is different for the two grades. 'White grade' material has only a slight decrease in strength in clear contrast to 'black grade' material which demonstrate a very strong dependence. This result seem to be caused by a high macroscopic compressive stress in the diamond, especially on the nucleation side. This explanation is supported by the height profiles (measured by a laser topography) of discs from wafer b15, b19/20/21 and b16 which showed convex bowing of the nucleation sides, which is reduced with increasing thickness.

4.3. Weibull Parameter

In FIGURE 6 the strength of a large amount of discs (N = 23), which were laser cut from 3 wafers grown under identical conditions, is plotted in Weibull representation. The strength values for the N samples were sorted in ascending order and the cumulative fracture probability $F_i = (i - 0.5) / N$ (for i = 1,..., N) was calculated. Describing the strength by a two parametric Weibull distribution, the Weibull parameters σ_0 and m can be determined with the maximum likelihood method[10]. In comparison to the high quality alumina (Deranox, thickness: 260 ± 5 μm), CVD diamond exhibits a higher strength σ_0 and also an extremely high m parameter, indicating a much narrower distribution of the strength data compared to many conventional ceramics.

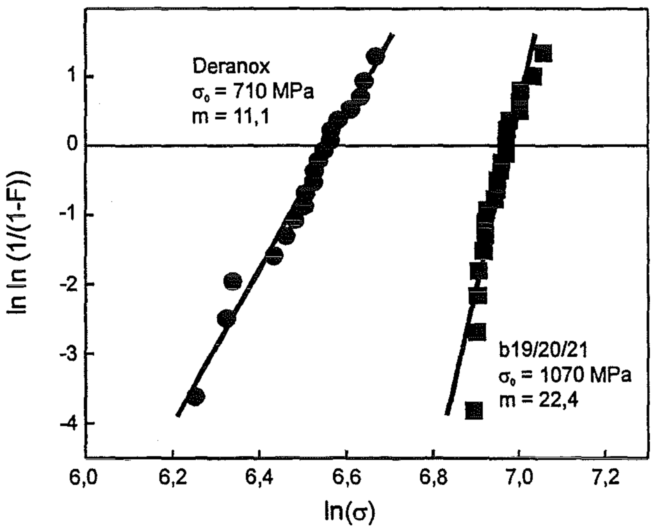

FIGURE 6
Weibull diagram for CVD diamond (growth face towards the ring) and high quality alumina.

5. CONCLUSIONS

Free-standing polycrystalline diamond wafer were grown to different quality by Microwave Plasma Assisted Chemical Vapour Deposition and partly further surface processed (lapping and polishing procedures). Two diamond qualities were identified: 'white grade' material for optical and radio frequency applications and 'black grade' material for mechanical and thermal management applications. Disc which were laser cut from these wafers were tested for their fracture strength using the ball on ring test. It was found that the median bending strength in comparison to a standard ceramic material like alumina is high and also the Weibull modulus m for a given quality was determined to be very high (m = 22.4). For the design of components out of bulk diamond the observed reduction of the strength with increasing thickness as a result of the gradient in microstructure have to be considered. Using the nucleation side of a diamond component in tension the resistance against spontaneous failure of the component is observed to be higher. The reason for this is not entirely clear but could be related to either the micro-crystalline nature of the nucleation surface which may resist the onset of the formation of micro-cracks which, when they reach a critical size, can rapidly propagate or the high compressive stress in the nucleation layer may have first to be overcome before the macroscopic tensile (bending) stress causes a crack to develop. Distinct differences were found in the measured strength and the ratio of transrystalline and intercrystalline crack propagation for the two qualities. These differences my be caused by the coarser microstructure (including critical defect distribution) in 'white grade' diamond, the different texture in combination with twinning, internal stresses or even differences of grain boundary strength, requiring further detailed investigations.

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