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Mechanical strength of neutron-irradiated window materials

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Abstract

Highly specialised window materials considered for transmission lines in plasma heating and diagnostic systems in nuclear fusion reactors were studied in terms of mechanical strength properties and potential radiation effects introduced by neutron irradiation up to 10^{21} n/m^2 ($E > 0.1$ MeV). Small disks of CVD diamond cut from model windows for high power transmission and from rods of a special fused silica grade (KU1) with radiation-hard optical properties were tested together with disks of commercially available high quality silica grades. Based on a biaxial mechanical test method, the influence of specimen machining could be kept under special control. The results obtained for CVD diamond clearly indicate that median strength values of 400 MPa and high Weibull moduli of 20 can be maintained with structural damage introduced at 10^{-4} dpa. For high quality silica grades, median strength levels of 300 MPa were reached in the test geometries applied. However, they tend to be very sensitive to changes in the surface quality. Median values of about 120 MPa and Weibull moduli of 10 can be taken for conservative strength evaluations for spontaneous failure.

1. Introduction

In electron cyclotron wave systems for heating and current drive (ECH-CD) and for plasma diagnostics, special window structures have to fulfil vacuum operation and tritium retention requirements with ideally broadband transmission and low power absorption. Actual window materials [1], which are CVD diamond for high power transmission and fused silica for broadband transmission, imply a potential use over a wider spectral range reaching from DC/RF applications to optical systems. Material performance analysis requires mechanical strength studies complementing the physical database, which is focused on dielectric and optical absorption as well as on thermal conductivity. Structural damage induced by neutrons potentially degrades the material properties. Whereas mechanical strength in fusion ceramics like alumina and SiC was investigated in experiments reaching dpa levels [2], for CVD diamond and fused silica neutron irradiation studies were missing even at the 10^{-4} dpa level which is considered as a conservative testing condition for window materials.

2. Parameterisation of window behaviour under mechanical loads

The mechanical strength properties of all potential window materials is characterised by their brittle behaviour. This means because of their purely elastic behaviour, that a window will bend when an external load (F_{ld}) is applied until a critical (tensile) stress value (σ_c) is reached. Then window failure will result from spontaneous crack formation. The amplitude of the maximum bow (b) and the maximum of the tensile stress (σ_{max}) depend on the window geometry, the distribution of the load and the rigidity of the window fixture [3]. For a disk (radius R, thickness t), the expressions for the various scenarios can be systematised by using the following expression:

$$b^* = \frac{b}{t} = gA^4 \frac{p}{E}, \quad (1)$$

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Table 1
Analytical expressions of the boundary factors g and g^* which factorise the expressions for the maximum bow and stress, cf. Eqs. (1) and (2), of a transmission window under external load for special loading and fixture conditions (based on Ref. [4])

	Rim rigidly clamped; subjected to uniform load over full radius R	Rim simply supported; subjected to uniform load over full radius R	Rim simply supported; subjected to concentrated load over restricted radius R_{ld} ($\ll R$)
g	$(3/16)(1-\nu)(1+\nu)$	$(3/16)(1-\nu)(5+\nu)$	$(3/4)(1-\nu)(3+\nu)$
g^*	0.75 (at rim)	$(3/8)(3+\nu)$ (at centre)	$(3/2)[1+(1+\nu)\ln(R/R_{ld})]$ (at centre)

$$\sigma_{max} = g^* A^{-2} p. \tag{2}$$

In these expressions, factorisation is achieved which contains dimensionless 'boundary factors' g and g^* that fully describe the loading and fixture scenario. The window geometry enters by a separate factor based on the 'aspect ratio' A $(= t/R)$. The applied load is normalised to an equivalent pressure p $(= F_{ld}/(\pi \cdot R^2))$. The elastic properties of the material enter through the Young modulus E into Eq. (1), and the Poisson number ν into g and g^*. For various limiting cases, the boundary factors are available in analytical form (cf. Table 1) [4]. For specific configurations of transmission windows, they can be obtained by applying the basic expressions to describe the experimental data and the finite element modelling of pressurised windows.

In the elementary view of fracture mechanics, spontaneous failure occurs when the following condition is fulfilled that allows a crack ('flaw' with a tip radius ρ) to propagate:

$$\sigma_c = K_{Ic}(Y\sqrt{\rho})^{-1}, \tag{3}$$

where the fracture toughness (K_{Ic}) is a material constant and Y a geometrical factor in the order of 1 [5,6]. In this view, the irradiation effect on mechanical properties should either affect directly the fracture toughness or the critical flaw size or indirectly modify the crack propagation [1].

With a typical ρ^{-x} distribution function for the flaw size [5], the special weight of the largest flaws, cf. Eq. (3), leads to a pronounced distribution of the observed critical stresses (σ_c) in window materials. The related failure probability (F) of a mechanically stressed window is described by the cumulative Weibull function:

$$F(\sigma_c) = 1 - \exp\left(-\frac{\sigma_c}{\sigma_0}\right)^m. \tag{4}$$

The Weibull parameters required for calculating the failure probability of a component according to Eq. (4) are the median strength (σ_0) and the Weibull modulus (m). The essential role of the Weibull parameter becomes obvious when results from destructive testing of (small) specimens have to be used in designing (large) components. Depending on the origin of flaws being mainly in the bulk or at the surface, the probability of reaching critical flaw sizes increases with the effectively stressed surface (S) or volume (V). Accordingly the reference strength value has to be scaled according to

$$\sigma_S = \sigma_0(S/S_0)^{1/m} \quad \text{or} \quad \sigma_V = \sigma_0(V/V_0)^{1/m}, \tag{5}$$

where the Weibull parameters as well as the scale parameters (S_0, V_0) relate to the test conditions.

Actual window design generally introduces safety factors (f_S) before assigning the geometrical parameters based on allowable maximum stresses, cf. Eq. (2) [7,8]. Two obvious terms depend on the Weibull modulus which assess the margin ($f_{S,F}$) defined by the maximum permissible failure probability (F_p) and the margin ($f_{S,V}$) defined by volume or surface scaling:

$$f_{S,F} = F_p^{-1/m} \quad \text{and} \quad f_{S,V} = (S(V)/S_0(V_0))^{1/m}. \tag{6}$$

Additional factors may arise from uncertainties in the median strength data or from grown-in stress which shows up in an effective bow in absence of applied loads.

3. Experimental

3.1. Materials

CVD-diamond grades suited for high power transmission windows are available as large area windows from DeBeers (Charters, UK) and the FhG-IAF (Fraunhofer-Institut für Angewandte Festkörperforschung, Freiburg, Germany) [9]. For the destructive strength studies, specimens were acquired which were laser-cut at FhG-IAF as 11 mm disks from typical window sized disks grown from the 'Ellipsoidal Reactor' approach [10]. Accordingly three distinct specimen sets were studied: specimens ER4_62 (#1–20) originated from a 4 in. window (1.0 mm thick), ER6_55 (#1–11) from a 2.5 in. window (0.9 mm thick) and ER6_56 (#1–10) from a 2.5 in. window (1.5 mm thick). From previous investigations on the mechanical properties of CVD diamond it was known that there is a marked difference in the strength of the fine-grained nucleation face and the coarse-grained growth face; therefore the more conservative test with the growth side in tension was consistently chosen. Surface roughness was below the resolution limit of the laser topography method used for quantifying the surface quality ($R_a < 0.1$ μm). Effects of the surface quality were not considered as the older studies [8] had already pointed out that critical flaws originated from bulk defects rather than surface defects.

KU1 quartz glass provided by the Russian Federation within the ITER task sharing agreement has been shown to be highly radiation resistant with respect to its optical properties for use in both diagnostic and remote handling applications [11]. From 16.5 mm diameter rods, specimens were cut within the framework of the European Fusion Development Agreement at CIEMAT. It was important to get specimens with a sufficient parallelism (thickness variation less than ± 10 μm for thickness of 0.8–1.0 mm). This was only achieved with machining techniques standardly used for ceramics. As it is well known for glass [7] that the surface quality is a major strength limiting factor, the machined faces were tested which had a well-defined surface quality ($R_a \approx$ 1.3 μm). Representative for the commercially available high purity silica grades which are already in use in EC wave diagnostic systems, the grade 301 of Infrasil from Heraeus (Hanau, Germany) was selected which is free of any light-scattering gas bubble content. The specimens (11 mm diameter) were studied with an as-received optical surface quality ($R_a < 0.1$ μm) and a abrased surface ($R_a \approx 1$ μm) to assess the degree of strength reduction due to machining in these special glasses. A number of 8–10 specimens were available in both surface qualities as neutron-irradiated specimens as well as control specimens.

3.2. Neutron irradiation

The mechanical strength studies are focused on the aspect of spontaneous crack growth. Subcritical crack growth (SCCG) in alumina and silica grades which has been previously studied under ionising γ-radiation and an applied electrical field by Pells and Boothby [13,14] and which is dependent on environmental conditions is beyond the scope of this work, as it is targeted at clarifying a potential permanent radiation effect induced by structural damage.

For this purpose specimens were neutron irradiated at GKSS (Geesthacht, Germany) at 320 K in a fast neutron flux of 4×10^{15} n/(m^2 s) ($E > 0.1$ MeV) in a Cd-screened capsule to effectively reduce the thermal neutrons. The achieved neutron fluence was 10^{21} n/m^2 which corresponds to a structural damage level of 10^{-4} dpa which is a conservative upper level expected for windows under ITER conditions with no direct plasma sight.

3.3. Mechanical testing

The 3- and 4-point bending tests which are widespread techniques for measuring the strength of glass and ceramics have the significant disadvantage that it is difficult to eliminate undesirable edge failures. This problem is overcome by biaxial flexure tests [15] which was established here in the form of a ball-on-ring test. The specimen which is simply supported by a slightly smaller ring ($R_{rg} = 5$ mm for 11 mm diameter specimens, and 7.5 mm for 16.5 mm diameter specimens) is loaded by a ball with half the radius of the ring. The critical stress can be calculated on the grounds that the ball produces a uniform loading over a central area with $R_{ld} \approx t/3$ [15]. To the basic formula, cf. Eq. (2), a small correction has to be applied for the 'stiffening effect' caused by $R = R_{rg} < R_{disk}$ [16]. For data evaluation, the following data for the elastic parameters were taken: CVD diamond: $E = 1050$ GPa, $\nu = 0.1$ [12]; special silica grades: $E = 72.5$ GPa; $\nu = 0.17$ [17].

The loads were applied by a standard testing machine (UTS), they were measured in a load gauge located above the fixture of the ball. They were in the order of 30–90 N (KU1), 50–150 N (Infrasil 301) and 200–500 N (CVD diamond). Special considerations were given to the push speeds. They were set to 25 μm/s which proved to induce spontaneous crack formation within 1–5 s. This speed is considered to be fast enough to avoid possible systematic strength reductions by SCCG phenomena.

Two different methods to parameterise the data sets were used. In cases where sufficient numbers of specimens could be tested (typically six or more), the Weibull parameters were calculated the maximum likelihood method. Thus the median values were obtained for all irradiated specimens, the apparent Weibull modulus was reduced by the correction factor for small specimen numbers [5]. Confidence intervals showed a typical span of ± 3 for $m \approx 10$ and of $\pm 15\%$ for σ_0. For graphical representations, the cumulative failure probability was calculated using the expression $F_i = (i - 0.5)/N$ with i the number of the strength value in data set sorted in ascending order. For small specimen sets, which were the control sets for CVD diamond and KU1 material, only the average strength value was taken as representative for the measurement. Comparing the median and the mean value of the control specimens for ER6_56, good correspondence was found ($\sigma_0 = 388$ MPa, $\sigma_{av} =$ 378 MPa).

4. Results and discussion

4.1. CVD diamond

The median strength values of the neutron-irradiated specimens from all three CVD diamond windows (ER4_62, ER6_55 and ER6_56) all group between 400 and 440 MPa (cf. Fig. 1). Even though the various model windows covered a significantly wide thickness range (0.9–1.5 mm), no systematic thickness dependence was found. Such dependence could be expected from the

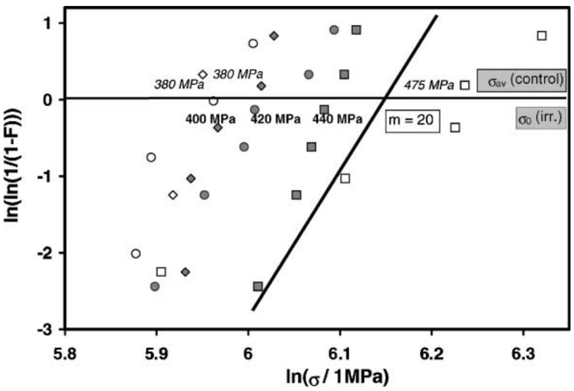

Fig. 1. Weibull analysis of the critical strength measured in the three specimen sets of CVD diamond after neutron irradiation to 10^{21} n/m^2 ($E > 0.1$ MeV) (full symbols): ER4_62 (squares), ER6_55 (circles) and ER6_56 (diamonds). Inserted numbers indicate the median strength values. The straight line represents a line with a slope given for Weibull modulus of ($m = 20$). For the unirradiated control specimens (open symbols), only the mean value of the measured strength data are indicated due to limited specimen numbers.

grain coarsening effect put to evidence for these CVD diamond grades (mainly for disks of much smaller thickness) [8,12,18]. The finding is especially obvious for the results obtained for the 4 in. window (ER4_62) where the median value (440 MPa) is clearly larger than the one observed for window ER6_55 (400 MPa) despite of a similar even somewhat smaller thickness. The values obtained agree well with values extrapolated into this range using the results of Refs. [8,18] which imply a range of 370–450 MPa. This assessment is clearly corroborated by the mean strength values obtained with the control specimens from the 2.5 in. windows which group at 380 MPa. There is a startling difference observed for the control specimens from the 4 in. window which show a larger average strength (475 MPa).

The Weibull modulus m is found to range around 20, as Weibull analysis yields values between 17–35 (uncorrected) and 13–26 (corrected). These values are in perfect agreement with values observed in systematic studies of different CVD diamond grades which state that above a certain state of grain coarsening (which is typically given in disks larger than 0.2 mm for the present grades), a high Weibull modulus ($m = 20$) is found [18]. Obvious deviation is again found for the unirradiated control specimens of ER4_62.

The major source of critical flaws is attributed to 'microcracks' which form as a consequence of internal stresses built up in the microstructure related with the columnar growth process [8,18]. The present results indicate that the structural damage introduced at a 10^{-4} dpa level is not effective in significantly modifying this strength limiting factor.

The apparent larger strength values in the unirradiated control specimens of the 4 in. window are not judged to contradict to this view. Rather these data are interpreted as an indirect evidence for particularly grown-in stresses during the growth of this particular window. Bowed large area CVD diamond windows are known for which the growth face is found to be under compressive strain. Such irregular stress term would increase the measured critical tensile stress required for crack formation. This systematic stress contribution appears to be not as relevant for the irradiated specimens which just may resort to the individual sampling or less likely to stress relaxation induced by the neutron irradiation.

4.2. Silica

The ball-on-ring tests performed on the Infrasil specimens and their Weibull analysis document a pronounced variation of the strength properties on the surface quality (cf. Fig. 2). When the surface under tensile load is tested with the as-received optical quality outstanding levels of the median strength are found (300 MPa) which exceed by far values specified by the producer (67 MPa) and those of similar grades (60 MPa) by Dynasil (Berlin, NJ, USA). For the abrased surfaces median values are found which are still a factor of 2 larger than the specifications. The increase in Weibull modulus from $m = 5$–8 to 10 indicates that the critical flaws at the surface are not only larger in the abrased specimens but also more homogeneous. Clearly the neutron-irradiation does not affect the strength parameters of the abrased specimens. The strength reduction for the case of optical grade surfaces is significant

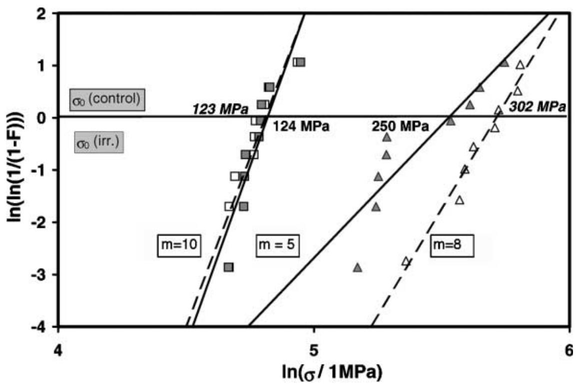

Fig. 2. Weibull analysis of the critical strength measured for disks of Infrasil 301 with abrased surfaces (squares) and with surfaces of optical quality (triangles). The data sets are quantified in terms of the median strength (σ_0) and the Weibull modulus (m) for neutron-irradiated specimens (full symbols) and for unirradiated control specimens (open symbols).

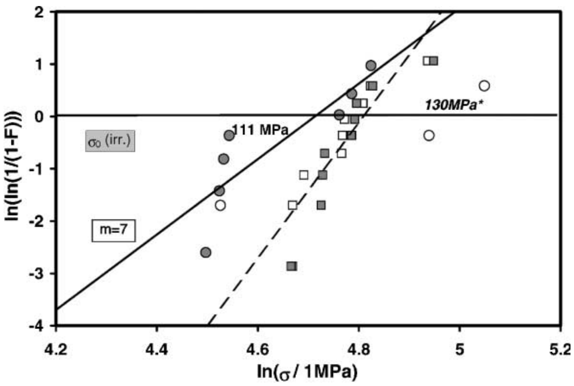

Fig. 3. Weibull analysis of the critical strength measured for disks of KU1 301 with machined surfaces. The data for the neutron-irradiated specimens (full circles) are parameterised with a median strength of 111 MPa and a Weibull modulus of $m = 7$. For the unirradiated control specimens (open circles) only the mean value of three strength data is given. For comparison the strength data for Infrasil with abrased surface (squares) from Fig. 2 are included.

(250 MPa), yet it is likely to fall within the extent inherent to the variability of the surface flaws due to external handling. The very low value of the Weibull modulus observed here ($m = 5$) appears to be only an approximate number describing a bimodial distribution, saying that some specimens exhibit surface flaws like in the as-received material and others have larger flaws introduced.

The results obtained with the KU1 specimens (cf. Fig. 3) that are tested with the surface quality resulting from the machining process group perfectly around the median strength values of the abrased surfaces in Infrasil (124 MPa). The neutron-irradiated specimens show some smaller strength (111 MPa) and larger scatter which, however, in the light of the observations made with the Infrasil specimens only hint at a less homogeneous surface quality. This also implies that the slightly larger mean values obtained for the few control specimens fall within the uncertainties of the sampling.

For all the measured sets of strength data, the predominance of the surface flaws over the structural damage induced by neutrons at a level of 10^{-4} dpa is obvious. The results even indicate that there is a fair chance that normal specimen handling in the course of an irradiation project could mask potential contributions from preformed volume (or surface) defects in high quality silica disks.

5. Conclusions

Based on an biaxial mechanical test method, the influence of specimen machining could be kept under special control for strength studies on neutron-irradiated high performance materials. The results obtained for CVD diamond clearly indicate that median strength values of 400 MPa and high Weibull moduli of 20 can be maintained in disks from model windows with geometries applicable in transmission systems for plasma heating or diagnostics. Structural damage introduced at 10^{-4} dpa induces apparent degradation. For high quality silica grades, median strength reach levels of 300 MPa in the test geometries applied. However, they tend to be very sensitive to changes in the surface quality. Median values of about 120 MPa and Weibull moduli of 10 can be taken for conservative strength evaluations for spontaneous failure in transmission components based on Infrasil (301) and KU1 grades. This data sets are also applicable in the case of neutron irradiation up to 10^{21} n/m^2 ($E > 0.1$ MeV).

With the finding that CVD diamond is characterised by volume flaws and high quality silica by surface flaws, the measured Weibull parameters can be used to evaluate tolerable critical tensile stress values following the approach proposed in Section 2. For instance, the median strength value for CVD diamond must be reduced for a tolerable failure probability $F_p = 10^{-3}$ and an effectively stressed volume in the component, which is 10 times larger than in the test specimen, by a safety factor of 1.5 (where the volume scaling gives a minor factor). The actual safety factor typically applied (3–4), however, includes an additional safety margin for grown-in stresses. Applying the same considerations to silica, the safety factor would be 2.5 and thus the critical stress value reaches the typical specifications of the commercial producers of these grades.

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