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Solid-state diffusion bonding of glass-metal for the International Thermonuclear Experimental Reactor (ITER) diagnostic windows

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Abstract

The United Kingdom Atomic Energy Authority are involved in the design and manufacture of the diagnostic windows for ITER. ITER is an international project, with 35 nations collaborating to design, construct and operate a prototype controlled nuclear fusion reactor in southern France. As well as providing line of sight for diagnostics, the windows also form part of the reactor primary containment boundary and are consequently classified as nuclear Safety Important Class 1 (SIC-1) components. The windows will be the first SIC-1 components in the world which are non-metallic. The current manufacturing process involves diffusion bonding a glass window to an Inconel 625 ferrule via an aluminium interlayer. This report discusses this diffusion bonding process and details the specific challenges related to component qualification for the intended nuclear SIC-1 application.

Keywords: Diffusion bonding, Ceramic-metal joining, Diagnostic windows, Nuclear fusion, ITER

1. Introduction

The United Kingdom Atomic Energy Authority are involved in the design and manufacture of the diagnostic windows for the International Thermonuclear Experimental Reactor (ITER). ITER is an international project, with 35 nations collaborating to design, construct and operate a prototype controlled nuclear fusion reactor in southern France. A CAD model of the ITER machine is illustrated in Figure 1(a).

The ultimate goal of the ITER device is to produce 500MW of fusion power with a ten-fold return on input energy (i.e. produce 500MW with a 50MW input power). In order to achieve this goal, a mixture of deuterium and tritium will be required in the vessel. As a result, the ITER site is considered to be a basic nuclear installation (INB No 174) as defined in French law [1] and as such is regulated by the French Nuclear Safety Authority (ASN). The ASN's Article INB No 174-23 states that the first confinement boundary shall limit the size of leaks and provide a means for capturing any leaks and passing them into the detritiation system [2].

As ITER contains several diagnostics which require line of sight (at various wavelengths) to the plasma, it is necessary to include some "windows" manufactured from non-metallic materials into this first containment structure [3]. The current catalogue of standard diagnostic window assemblies offers nine configurations of fused silica windows in various sizes with nominal clear view diameters varying from 25 mm to 160 mm (a maximum surface of 0.02 m^2 is defined in the requirements). A CAD model of a typical ITER diagnostic window assembly is illustrated in Figure 1(b). As these windows form part of the containment boundary they are classified as Safety Important Class 1 (SIC-1) [3]. However, conventional pressure vessel codes are not applicable to brittle materials [4]. Furthermore, the transparent element, diffusion bond and ferrule are considered to be outside the scope of the contemporary design codes (e.g. RCC-MR [5] or ASME VIII [6]).

This report discusses the diffusion bonding process and then details the specific challenges related to component qualification for the intended nuclear SIC-1 application.

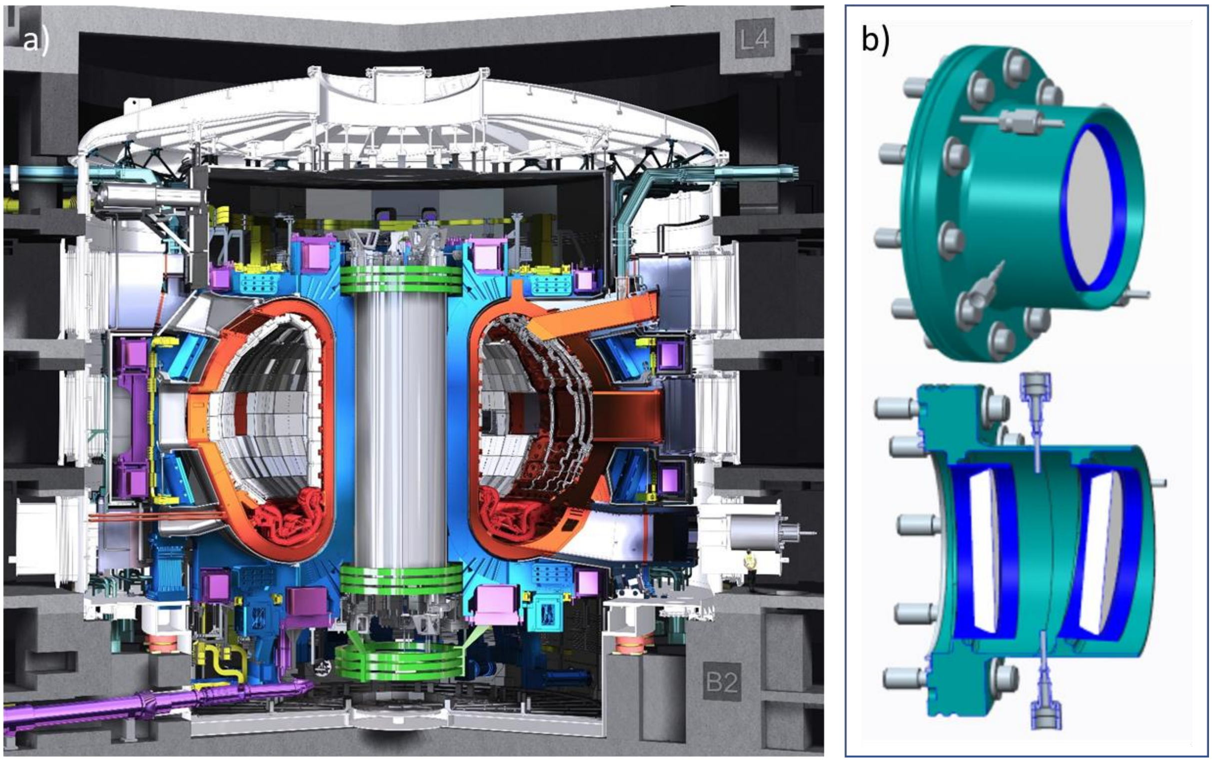

Figure 1: CAD models of, (a) the ITER tokomak and (b) the ITER diagnostic window assemblies.

2. Diffusion bonding process

2.1. Overview of the process and materials

Figure 2(a) presents a CAD model of the key components and material configuration concerned in the diffusion bonding process. Specific details concerning the process parameters is commercially sensitive and as such only a high-level overview of the process will be discussed in this report.

An aluminium interlayer is used to bond a tapered fused silica optic into the matched taper of an Inconel 625 ferrule. Prior to the process, the aluminium in cleaned to remove contaminants. A bonding furnace is used to apply a large load and heat (50-60% melting temperature) to compress the aluminium into the joint. The bond is then held at temperature for diffusion to occur. Once cooled, a helium leak test is conducted on the assembly as a final acceptance test to confirm the bond quality. Figure 2(b) shows a photograph of the finished diffusion bonded window assembly.

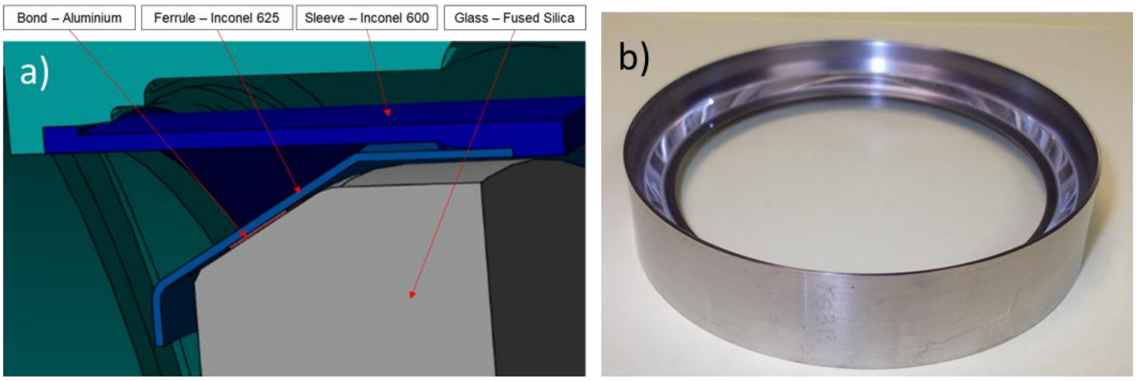

Figure 2: (a) CAD model illustrating key components associated with the diffusion bonding process, and (b) a photograph of the finished diffusion bonded window assembly.

2.2. Physico-chemical structure of the diffusion bond

A. Lunev [7] showed that elemental inter-diffusion causes physico-chemical changes to the substrate materials to form the hermetically sealed diffusion bond. Figure 3(a) shows a cross-section macrograph of the diffusion bond interface. A Tescan MIRA3 Scanning Electron Microscopy (SEM) system with Schottky field emission gun installed at the UKAEA's Materials Research Facility (MRF) was used to assess the interface at higher resolution. Secondary electron images presented in Figure 3(a) and (b) reveal that the Inconel 625/Al diffusion bonding occurs non-uniformly, with large variations of the interface layer thickness. For example, Figure 3(b) shows a clear interface layer thickness ~4 µm, whereas in Figure 3(c) the interface layer is almost indistinguishable.

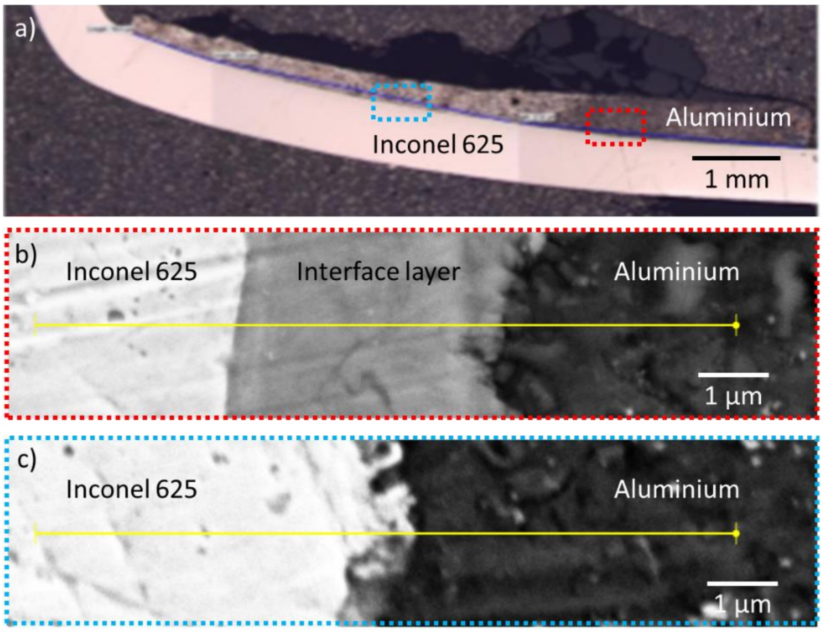

Figure 3: Microscopy analysis of the Inconel 625/Al diffusion bond showing, a) cross-section macrograph of the diffusion bond interface, b) secondary electron SEM image of the Inconel 625/Al diffusion bond with large interface layer, c) secondary electron SEM image of the Inconel 625/Al diffusion bond with small interface layer. The yellow lines in (b) and (c) depict the path of subsequent EDX line scans

Energy-dispersive X-ray spectroscopy (EDX) was used to measure the elemental migration across the interface. EDX analysis was performed using the Oxford Instruments EDX detector attached to the interface port of the SEM system. Spatial resolution of EDX is determined by the volume in which beam electrons interact with the material producing characteristic X-rays with a lower accelerating voltage (which defines the electron energy) providing a higher spatial resolution. To calculate the interaction volume of the electron beam at the Inconel 625/Al interface, CASINO [8] software was used. An energy of 9 keV was chosen which corresponds to 1 µm x 1 µm surface interaction area of depth 1.5 µm with ± 0.4-0.5 µm spatial resolution. Results of EDX line scanning across the interface denoted by the yellow line in Figure 3(b) is presented in Figure 4.

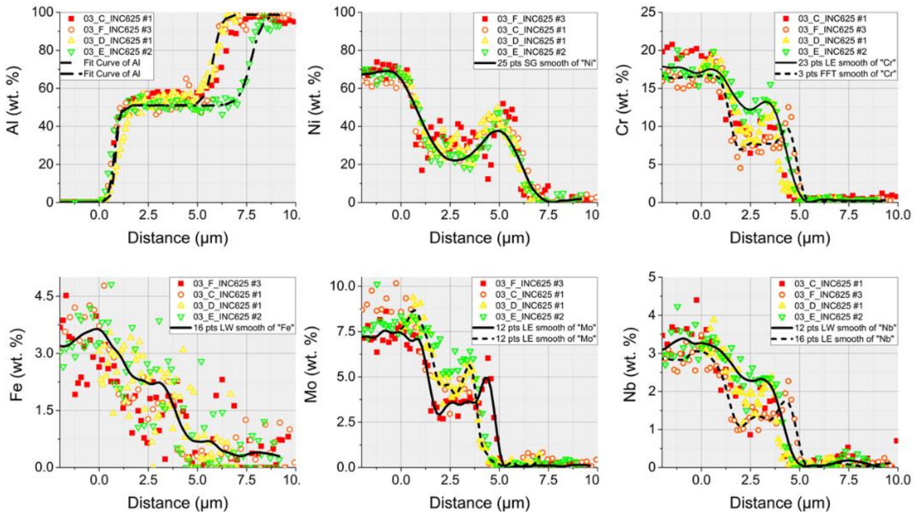

Figure 4: EDX line profile for key elements across the Inconel 625/Al diffusion bond interface defined by the yellow line in Figure 3(b). The colour markers denote the experimental scatter from seven EDX line scans, respectively. The standard function library in Origin Pro was used for the Boltzmann fit (black line) which depicts an average compositional profile.

Figure 4 shows an extended diffusion bond region with a thickness 7-10 µm. This extended region was perfectly visible in SEM secondary electrons in Figure 3(b). On the other hand, in those samples where two materials were demarcated by a visually sharp boundary (e.g. Figure 3(c)), the composition profile was much less diffuse. The sharpest composition profiles at the Inconel 625/Al interface showed an inter-diffusion layer between 0.8-3 µm.

Interestingly, the boundary layer thickness seems to be smaller for Cr, Mo, Nb, Fe (~1.2 µm) and larger for Al and Ni (~2.0 µm). This is due to the difference in their respective diffusion coefficients. Hence, even for the thinnest inter-diffusion layers, a region denuded of Cr, Mo, Nb, and Fe could be identified. The variation in Inconel 625/Al interface layer thickness is probably due to a variation of the diffusion coefficient caused by chemical heterogeneity or other factors, including the variation of stresses during diffusion bonding. Other factors such as texture, non-uniform temperature distribution during diffusion bonding or imperfect surface finish could also affect the thickness of the interface layer.

Similar analysis was performed for the SiO/Al interface, with the results presented in Figure 5. The thickness of the SiO/Al inter-diffusion layer was 0.75-2.0 µm. The SiO/Al boundary thickness is more consistent and sharper than the Inconel 625/Al interface. This indicates lower diffusivity and heterogeneity of the SiO/Al interface compared to Inconel 625/Al.

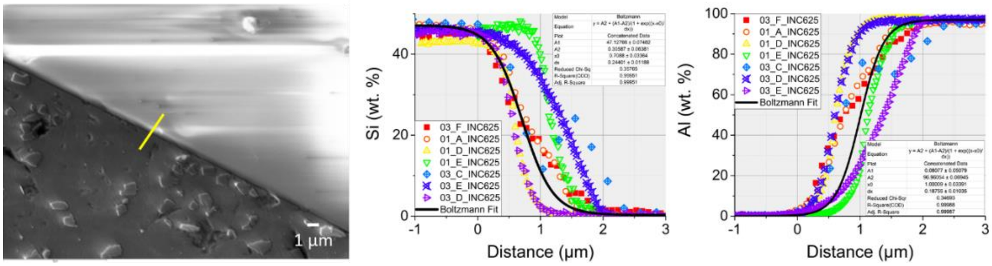

Figure 5: Microscopy analysis of the SiO/Al diffusion bond showing, a) secondary electron SEM image of the SiO/Al diffusion bond with small interface layer, (b) EDX line profile for Si, and (c) EDX line profile for Al. The yellow line in (a) depicts the path of subsequent EDX line scans in (b) and (c). The colour markers denote the experimental scatter from seven EDX line scans, respectively. The standard function library in Origin Pro was used to for the Boltzmann fit (black line) which depicts an average compositional profile.

3. Qualification of glass-metal diffusion bonds for nuclear SIC-1 application

Several issues present themselves regarding qualification of glass-metal diffusion bonded components for nuclear SIC-1 application. The most prominent of these issues being the fact that glass is a brittle material which has never been qualified for a nuclear SIC-1 application and that diffusion bonding is not a recognised manufacturing process in any contemporary design code. Nevertheless, without these non-metallic diffusion bonded viewports providing line of sight into the plasma for diagnostics, ITER would not be able to operate. Therefore, it is evident that a certain amount of pragmatism from all parties is required in the endeavour towards SIC-1 qualification. Key issues related to the future qualification activities will be discussed hereafter, based upon the work of R. Bamber [9].

3.1. Issues related to the qualification of the ceramic

3.1.1. Ceramics are inherently brittle

The amorphous atomic structure of fused silica, prohibits dislocation movement in the atomic structure, rendering the material brittle. Typically, a fracture toughness of approximately 0.75 MPa√m is observed in fused silica [10]. The theory of linear-elastic fracture mechanics allows us to define a critical crack size for each material (with its inherent fracture toughness) in a given stress field, above which it will fail catastrophically.

Traditionally, design of brittle materials utilises either probabilistic or deterministic methods. These methods typically exploit Weibull statistical methods or selective proof testing. Probabilistic methods demonstrate, through mathematical models of the flaw distribution, that a certain target probability of failure in service (typically 10^{-6}) is not exceeded. Deterministic methods use a proof testing procedure in order to demonstrate (via an overload condition) that a flaw of the critical size is not present throughout each component destined for service, provided that these survive the proof test.

The brittle nature of ceramic materials implies that gross movements are accommodated by crack growth as opposed to plastic deformation. It is interesting to note that micro-cracking of materials may not cause leaks greater than 10^{-2} mbar.l/s, which is thought to be acceptable from a safety and operational perspective until the defective window is replaced. It should also be noted that stress fields caused by secondary (displacement driven) loads may cause only insignificant cracking if the crack growth outruns the stress field (i.e. the stress field is relieved by crack growth) [11]. However, for these arrested cracks to be considered insignificant they must neither allow a significant leak from the torus nor affect the window's ability to survive the loadings sustained during the potential events (plasma disruption, seismic etc).

3.1.2. Sub-Critical Crack Growth (SCCG) phenomenon

An additional complication for the demonstration of structural integrity for the transparent element is the phenomenon of SCCG (or Static Fatigue) whereby cracks can grow under the application of tensile stress. This implies that cracks, which are not of the critical size at the beginning of life of the ceramic may grow during service, consequently reaching the critical size, leading to catastrophic failure. The theory describing SCCG is given in more detail in [12]. The reader should note that the issue of SCCG implies that the proof-test procedure itself weakens the service components.

3.1.3. Inherent variability of ceramic materials vs metallics

The qualification of ceramic materials via prototype testing is rendered difficult by the greater variability of strength when compared to metallic materials, making it problematic to infer production piece survival from prototype survival of a given loading [4].

3.1.4. Measurement or modelling of residual manufacturing stress on the ceramic

Although several research projects are ongoing in this area, at present it is not possible to determine with any accuracy the residual manufacturing stress in the ceramic. High stress gradients, combined with current computing limitations, render the accurate predication of the peak stress impractical in all but 2D modelling, itself questionable due to the inherent assumptions and the observed profile of bonded windows. This problem is exacerbated by the difficulty in generating a robust and traceable model for behaviour of the aluminium interlayer. Determination of material properties and stress states in a repeatable fashion is difficult due to the distributed material properties of ceramics and the singularity occurring at the ceramic/metallic joint in mathematical modelling (Figure 6), respectively. Finally, experimental measurement of residual stress in the non-crystalline material is not straightforward, with synchrotron X-ray pair distribution function analysis currently being explored as candidate method for direct measurement of residual stress in the ceramic.

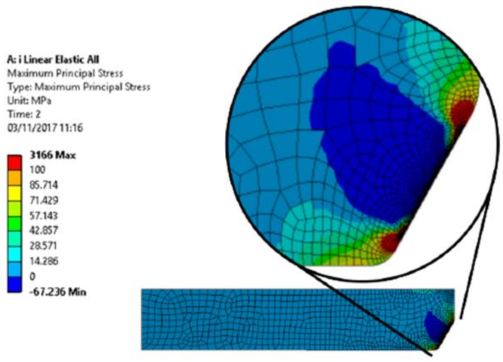

Figure 6: Linear elastic finite element model of the manufacturing residual stress in the ceramic at the diffusion bond interface. Peak stresses result from mathematic singularities.

3.2. Qualification of the diffusion bond

The bonding must be considered as a special process (analogous to welding) within the qualification of the window assembly. This process has been used successfully to create vacuum windows for Joint European Tours (JET) and other scientific experiments by the UK Atomic Energy Authorities Special Techniques Group for approximately 40 years.

Typically push out tests have been used to demonstrate the bond strength. Demonstrating the bond strength at the end of life conditions is novel to ITER. The obvious aging effect of radiation is an additional complication to any thermal aging and cyclic stress effects that should be quantified carefully.

A range of options are available for the end of life design justification ranging from development of mechanistic models to a fully empirical approach adopting the creation and aging of multiple full-sized samples. The options investigated are listed below;

- Predictive mechanistic model analytical qualification
- Use of cohesive zone models (analysis with supporting experimentation)
- Semi-empirical testing on both reduced and full-sized specimens
- Empirical type testing

Unfortunately, each method considered contains significant technical challenges. In particular, it is difficult to determine the bond strength after aging (thermal, irradiation, cyclic loading etc.) and a high number of samples are required due to the stochastic nature of failure. It is useful to note that, empirically, windows tend to fail in the ceramic material adjacent to the bond, relieving some of the scrutiny on this part of the design. Due to the difficulties encountered when attempting to apply any of the standard design options described, it has been suggested to create a novel methodology with the intention that the benefits of each methodology can be retained. This methodology, schematically illustrated in Figure 7, intends to determine a bond strength from push-out testing combined with leak rate testing for the as-built window. This strength will be modified via the use of knock-down factors to account for the window aging processes. These knock-down factors will be determined experimentally. A design strength will then be generated, allowing comparison to the loads predicted in finite element modelling. This generates an explicit design margin whilst accounting for cyclic loading and other factors without the need for simulated category IV load condition testing.

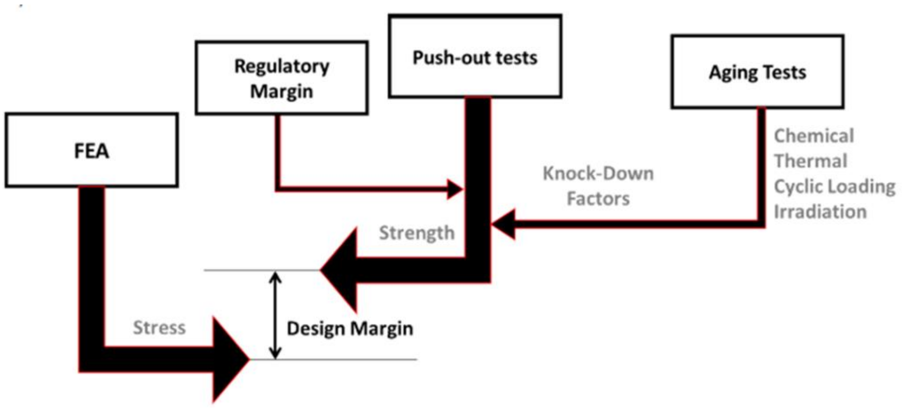

Figure 7: Schematic illustration of the proposed qualification route for the diffusion bond.

4. Conclusions

The processing and physico-chemical structure of an Inconel 625-Al-SiO diffusion bond is presented. Composition profiles at the inter-diffusion interfaces were measured with good spatial resolution (0.4-0.5 μm). The thickness of the diffusion bond interface layer at the Inconel 625/Al interface showed a large scatter (0.8-10 μm). The thickness of Al/SiO diffusion bond interface layer is much thinner (0.75-2 μm) with small variations (<1 μm) for all samples measured in this work. The effect of the variational diffusion layer thickness has yet to be quantified, but it will inevitably be a source of property scatter.

Issues related to qualification of the glass-metal diffusion bonded component for a nuclear SIC-1 application are discussed. Qualification of the ceramic disc is complicated by; i) inherent brittleness of ceramics, ii) sub-critical crack growth phenomenon, iii) inherent variability of ceramic materials vs metallics, iv) difficulty in measuring or modelling residual manufacturing stress. A novel methodology is proposed for qualification of the diffusion bond due to difficulties encountered when attempting to apply any standard design options. Work is ongoing in all these aspects.

5. Acknowledgement

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6. References

[1] *French Décret no 2012-1248 of 9th November 2012*. .

[2] *Décision no 2013-DC-0379 de l'ASN of 12th November 2013*. .

[3] P. Maquet *et al.*, "Development of ITER diagnostic window assemblies," *Fusion Eng. Des.*, vol. 88, no. 9–10, pp. 2641–2645, Oct. 2013.

[4] R. Bamber, R. Morrell, C. Waldon, and M. Shannon, "Design substantiation of ceramic materials on fusion reactor confinement boundaries," *Fusion Eng. Des.*, vol. 125, pp. 283–289, Dec. 2017.

[5] *RCC-MR 2007 Design and construction rules for mechanical components of nuclear installations*. 2007.

[6] *ASME Boiler and Pressure Vessel Code, Section VIII*. 2019.

[7] A. Lunev, "Determination of the composition profi les in the diffusion bond area of Ta/Al/SiO and INC625/Al/SiO joints," *UKAEA Internal Report*, 2018.

[8] H. Demers *et al.*, "Three-dimensional electron microscopy simulation with the CASINO Monte Carlo software," *Scanning*, vol. 33, no. 3, pp. 135–146, 2011.

[9] R. Bamber, "Report proposing a route forwards for the qualification of the bonds and windows," *UKAEA Internal Report*, 2019.

[10] J. Salem, "Fused Silica and Other Transparent Window Materials," in *40th International Conference and Expo on Advanced Ceramics and Composites*, 2016.

[11] M. Wachtman, J. B., Cannon, W. R., & Matthewson, *Mechanical Properties of Ceramics*, 2009.

[12] S. M. Wiederhorn, "Subcritical Crack Growth in Ceramics," in *Fracture Mechanics of Ceramics: Volume 2 Microstructure, Materials, and Applications*, R. C. Bradt, D. P. H. Hasselman, and F. F. Lange, Eds. Boston, MA: Springer US, 1974, pp. 613–646.