Monitoring and Assessment of the Effect of Defects in Ceramics

G. Seifert, J. M. Hausherr

As even small single defects in ceramic components can initiate their failure during operation, with potentially dramatic consequences for the user, researchers worldwide seek to safely detect critical defects and assess their relevance for failure under load before a component goes into application. In this paper, we introduce an experimental technique for observing the initiation and growth of cracks under mechanical loading in situ by help of X-Ray Computed Tomography (CT) and describe timely concepts to assess the criticality of voids and other defects in the volume or at the surface of ceramic components by help of finite element analysis.

1 Introduction

Typical requirements for high-performance ceramics are high mechanical strength in connection with very good reliability against fracture below the specified load limit, or excellent resistivity against thermal shocks and/or large temperature gradients. All these parameters are more or less directly related to the presence of defects in the components.

While it is of course the goal of any producer to minimise the number of defects in their components or bring them ideally to zero, the reality of ceramic production comprises a lot of possible sources for inhomogeneity or voids. Even if the raw material is perfectly processed and does not have any agglomerates or inclusions, the following typical steps like forming, debinding and sintering can easily generate or enlarge small defects. Thus, particularly for critical applications like aviation or space flight, it is necessary to guarantee that any component for use in aircrafts or spacecrafts is either defect-free or alternatively has been secured to resist the expected load for the anticipated operation time.

An established way to get this security is to perform proof tests for each individual component. As, however, these proof test procedures are very expensive and time-consuming, any experimental technique or simulation method which provides the same information with less effort is highly desirable.

In this paper, we discuss timely examples for monitoring defects in ceramic components by imaging methods and assessing their effects on component performance. First,

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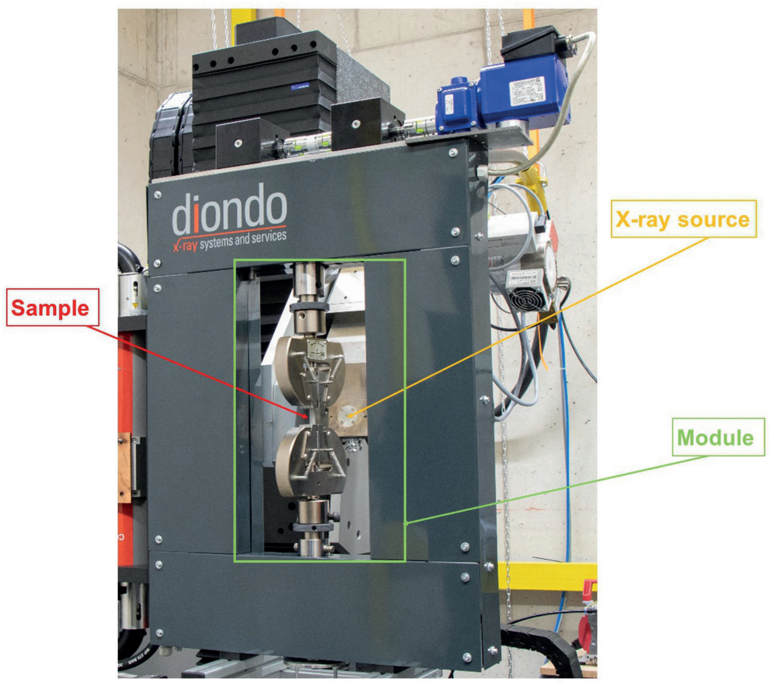

Fig. 1 Image of an in-situ setup with tensile module

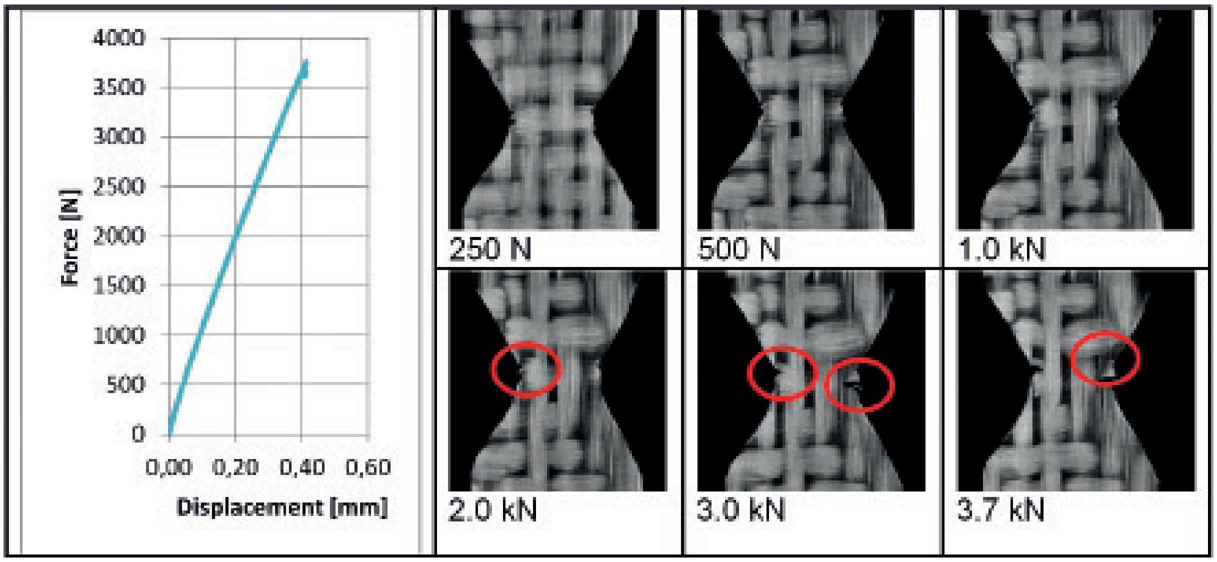

Fig. 2 Failure pattern of a GFRP sample under tensile load: measured force-displacement curve (left-hand side); and section images in the component at individual load levels (right-hand side)

a novel tool for observing initiation and growth of defects/cracks under mechanical loading in situ by help of X-ray Computed Tomography (CT) is introduced. Then, procedures to extract relevant volume or surface defects from CT, LSM (Laser Scanning Microscopy) or other imaging techniques into an FE (Finite Element) analysis for assessment of their criticality with respect to fracture under mechanical or thermomechanical load is described.

2 Computed tomography for in-situ analysis of local defect growth

Fraunhofer HTL, in cooperation with diondo, has developed a test frame for generic computer tomography systems. Using this test frame, components made of various materials including ceramics, can be examined volumetrically while they experience an additional thermomechanical or thermochemical load. The system is modular and can be mounted in basically any type of CT, complementing existing systems.

A special feature of the system is the possibility of a modular extension with various components. For example, in addition to a mechanical load (tension/bending/torsion and compression test), testing structures can also be simultaneously thermally loaded by integrating a climatic chamber for the range of –40 °C to +200 °C, or by implementing an oven for higher temperatures up to 1400 °C. Additional modules allow the controlled use of special atmospheric conditions including the simulation of (wet) chemical environments.

The CT measurements are performed at high resolution allowing to record volumetric images with a resolution up to 2 µm. Depending on the design, mechanical loads up to 150 kN are possible. Fig. 1 shows a test frame mounted in an existing tomography system for mechanical tensile testing up to 50 kN.

As an example of a simple tensile examination, Fig. 2 shows the measured force-displacement diagram of a glass fibre-reinforced polymer sample. The increase in tensile force causes elongation in the material, which, starting at around 2 kN, leads to a distortion of the fibre bundles in the matrix. Matrix failure begins around a tensile force of 3 kN, where first cracks are visible. With further increasing force, filament failure of individual glass fibres and, ultimately, component failure occurs at 3,7 kN.

3 Assessment of the criticality of defects by FE analysis

3.1 Volume defects monitored by CT

The in situ CT method described above is a very useful tool to observe the behaviour (potential growth) of individual defects in a ceramic component under a well-defined loading situation. While it appears well-suited to identify critical positions in components during their development as well as to deepen the insight about which kind of defects (considering shape, size, and position) are most dangerous for component failure, it is much less suited for a standard quality control of high-performance ceramic products. Main reason is that the critical inspection and evaluation of a time series of 3D-images currently has to be done by an experienced human operator, making the analysis time-consuming and costly.

Even without the in situ equipment, CT monitoring of volume defects in ceramics faces the basic challenge of balancing the total volume which can be scanned in one run against the spatial resolution achievable. As, depending on the material system, voids and other defects of 20–50 µm typical size and above can be relevant for the failure of even large components, the resolution of a CT scan has to be sufficient to safely detect such small voids or inclusions. This limits the maximum volume which can be accessed within the same scan to a typical dimension of several centimetres. For larger components, an intelligent strategy is required to find out the risk of component failure without the need to scan the whole component with high CT resolution.

A promising approach to identify security-relevant defects with strongly reduced effort and time is previous Finite Element Analysis (FEA). When the maximum thermal or mechanical load of a component during its application is known, the local distribution of stresses within a ceramic component can be determined precisely using current FEA software.

The often quite inhomogeneous distribution of stresses then allows restricting the CT scans to only few limited regions, where defects could cause the local strength limit to be exceeded. In the ideal case, i.e. when these regions are defect-free, the respective component can be released to application without further tests necessary. If the strongly stressed regions are not limited to a few critical positions, but cover larger parts of a component volume, several defects may be found requiring an individual assessment of their effects on the fracture limit.

Again, FE methods can be very helpful for this task: the typical procedure, as used by the Fraunhofer HTL, starts with the localisation of a defect and the definition of the void's 3D-surface, usually in form of a triangular representation. As visualised in Fig. 3, the defect shape is then transferred in digital form to an FE software package (here ANSYS), where it is used to generate a volume element (cube) containing the defect. The volume element is then being computationally exposed to external load, e.g. tensile strain like in Fig. 3, yielding the local stress distribution around the void. An example for such a volume-weighted distribution, normalised to the stress without void, is shown in Fig. 3 in form of a histogram.

Clearly the presence of the defect in the volume causes stress concentration extending to approximately a factor of 2 above the constant stress level expected in a perfect bulk material (represented by stress concentration factor of 1).

A reasonable estimate for the decrease of the local fracture strength can be obtained from the 95 % percentile of the stress distribution. In the example shown in Fig. 3, the 95 % percentile is located at a stress concentration of ≈1,45 (red vertical line); if the defect happens to be located within a region of maximum stress under load, one expects a decrease of the failure stress of the component by the same factor.

After a statistically relevant number of experiments for the verification of these numbers, the FE-based evaluation of the effects of defects is well-suited to qualify ceramic components for application. A further step towards automatized assessment could be achieved by including Artificial Intelligence (AI) algorithms into the procedure, both for the identification of defects in 3D-CT data and for the assessment of their severity for increasing the risk of component failure. This approach is a topic of current research at the Fraunhofer HTL.

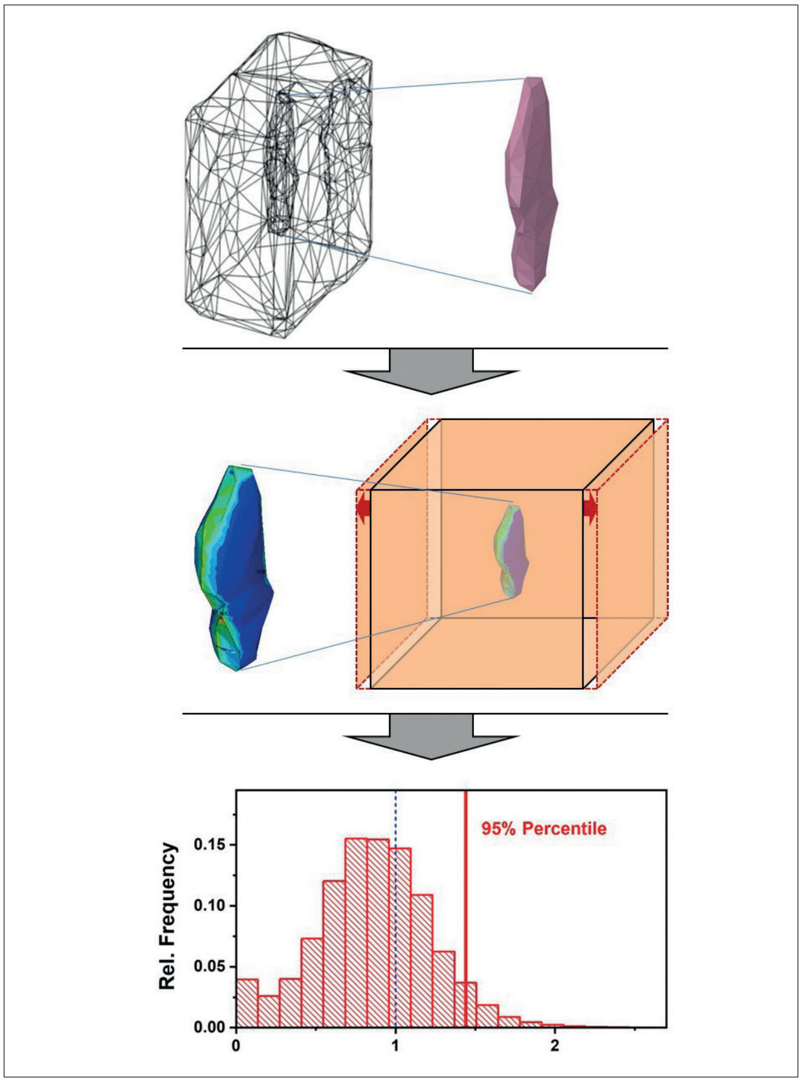

Fig. 3 Schematic overview of FE assessment of volume defects detected in CT scans of ceramic components

3.2 Surface defects monitored by LSM (Laser Scanning Microscopy)

As failure of ceramic components under mechanical loading (in particular tension or bending) is often initiated at the component surface rather than in the bulk volume, similar techniques to assess the impact of surface defects have been developed. In principle any imaging technique providing the surface topology of a ceramic sample can be used as experimental basis. There is, however, a tradeoff between spatial resolution and total area which can be scanned in one experiment.

For the purpose of evaluating defects on ceramic surfaces, modern optical techniques like LSM offer a very good compromise having submicron resolution on the one hand, and actually no limitation in the total area which can be scanned contactless on the other hand.

At Fraunhofer HTL, a combination of in-house and commercial software is used to assess ceramic surfaces for potentially fracture-initiating defects. The in-house algorithm was programmed to automatically select representative small sub-areas from

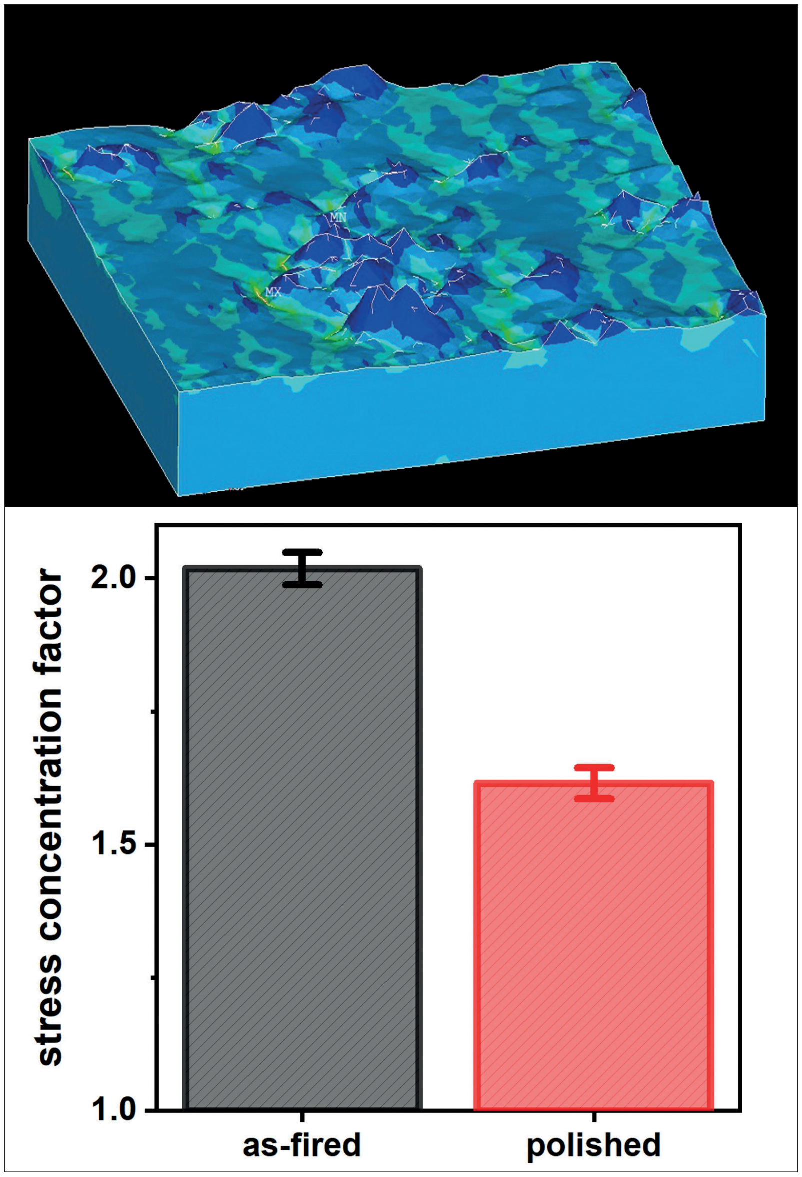

Fig. 4 Example for FE assessment of surface defects based on LSM measurements on alumina ceramics surfaces

LSM surface topology data for further analysis. In particular, a parameter for estimating the surface-specific stress concentration [1] is used to identify those sub-areas containing the potentially most "dangerous" defects with respect to limiting the fracture strength of a sample.

Several such areas are then converted into an FE mesh forming the surface of a volume element, which is subjected to an FE stress analysis under mechanical load – fully analogous to the technique for volume defects described above.

Figure 4 shows an example for a volume element generated from a 10 µm × 10 µm surface area measured with LSM on a polished alumina sample. The coloration of the surface represents the results of the FEA: regions with locally reduced stress (compared to the value for a perfectly flat surface) appear in dark blue, while local stress concentration is indicated by cyan via yellow to red colour. As tensile stress was applied, the largest stress concentration is observed within the grooves of the exemplary surface shown in Fig. 4.

For demonstration purposes the surfaces of alumina samples have been evaluated in two different states: (A) as-fired and (B) grinded and polished. For each case, five sub-areas have been selected, and their 95 % percentiles of stress in the volume elements close to the surface have been determined.

The mean values, as shown in Fig. 4, show clearly that the surface treatment reduces stress concentration in the surface grooves considerably (from ≈2,0 to ≈1,6, i.e. by approximately 20 %). The measured fracture strength of samples of type (B) increased by the same order of magnitude compared to samples of type (A).

4 Conclusions and outlook

The experimental and numerical techniques described in this paper are a promising collection of tools which can be used to identify and study locally the initiation and growth of defects in ceramic materials under load, as well as to assess their criticality with respect to component failure by computer simulations.

As the finite element analyses of individual defects can, with reasonable effort, only be done for a few locations within a larger component, an efficient selection of the relevant positions and, in particular, experimental validation of the numerical predictions is crucial, before this concept can prospectively replace proof testing.

Further work in this field will also include the use of AI algorithms for automatizing the analyses as far as possible and increasing their precision.

Reference

[1] Medina, H.; Hinderlitter, B.: The stress concentration factor for slightly roughened random surfaces: Analytical solution. Int. J. Solids Struct. **51** (2014) 2012–2018