ORIGINAL RESEARCH

The Acoustic Emission Testing in the Evaluation of Fracture Toughness of Brittle Materials

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Abstract

Aim: Evaluating the fracture resistance of dental ceramics is essential for assessing their behavior. This study aimed to validate a custom load-to-fracture test for assessing fracture strength compared to a conventional method.

Materials and methods: Acoustic emission testing, a non-destructive (ND) lab test, was employed to evaluate the fracture toughness (FT) of brittle materials by capturing sound waves generated by crack formation in failing samples. A total of 130 samples, divided into three types (glass sheets, zirconia sheets, and monolithic zirconia crowns), were tested. The fracture loads were measured using both custom and conventional methods.

Results: The mean fracture loads for glass sheets were 650.46 N ± 110.38 (custom) compared to 691.41 N ± 155.92 (conventional). For zirconia sheets, the values were 95.25 N ± 7.78 (custom) vs 112.75 N ± 31.26 (conventional). Monolithic zirconia crowns showed mean fracture loads of 1108.99 N ± 327.89 (custom) compared to 1292.52 N ± 271.42 (conventional). Statistically significant differences were evident in all three types, indicating lower values with custom testing for all samples.

Conclusion: The custom testing demonstrated an advantage in identifying cracks at lower loads, thereby enhancing the accuracy of fracture load values. Despite its limitations, the study suggests that the custom setup could be a viable alternative to conventional fracture load testing of brittle materials. However, further testing with more materials is recommended to enhance the results' accuracy and generalizability.

Clinical significance: The findings indicate that the custom load-to-fracture test can provide more accurate measurements of FT in dental ceramics, which is crucial for predicting their clinical performance and longevity.

Keywords: Acoustic emission test, Brittle ceramics, Fracture resistance, Monolithic zirconia, Sound harvesting test.

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Introduction

Ceramic materials are extensively used in dentistry due to their aesthetic qualities, making them suitable for dental prostheses, in particular, monolithic zirconia frequently chosen for its exceptional strength.[1] However, due to their inherent brittleness and limited tensile strength, ceramics are prone to failure under prolonged stress and varying forces,[2] making fracture resistance a key factor that significantly affects their lifespan and structural integrity.[3,4]

The precise detection of crack initiation, which marks the onset of failure in ceramic materials, is essential for accurately analyzing their stress response, particularly in terms of timing and the applied force. When stress is applied to ceramics, cracks originate from flaws known as discontinuities. These flaws or defects can be formed due to mechanical, chemical, or thermal processes. By increasing the load, the crack propagates until the component fails.[5]

To better understand this response, ceramics undergo rigorous fracture testing, such as FT tests, where materials are subjected to continuous stress to determine their fracture toughness (FT) and resistance values. The process evaluates materials by exposing them to simulated stress environments to measure their ability to resist crack growth and structural failure.[6]

Various destructive testing (DT) methods, such as strength and FT tests, are used to determine the performance of dental ceramics.

In the conventional FT test, a universal testing machine (UTM) with a spherical stainless-steel indenter applies a compressive load to the occlusal surface of the samples, to determine the force needed to induce fracture.[7]

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However, DT has several limitations. These tests destroy the samples, making them unusable for further testing. Moreover, DT offers a snapshot of the material's properties at a particular point of time, making it ineffective at detecting initial changes like the onset of cracks. Finally, DT provides a restricted assessment of failure modes, primarily focused on measuring peak failure strength, yet fails to provide a comprehensive understanding of the underlying failure mechanisms. This limitation can lead to FT estimation values which may exceed the true resistance values.[8]

Non-destructive testing (NDT) involves inspecting materials without causing damage. These methods play a vital role in assessing the structural integrity of materials across different industries, such as dentistry and ceramic production.[8,9]

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In the production of ceramics, NDT methods are vital for identifying structural defects without damaging the materials.[10,11] Common NDT techniques utilized for ceramic materials are radiographic testing, X-rays, computed tomography, penetration testing, ultrasonic testing, laser ultrasonics, infrared thermography, optical coherence tomography, and acoustic emission testing (AET).[12,13]

Acoustic emission testing is a well-established non-invasive technique used to detect stress waves generated within materials like dental ceramics when cracks initiate due to a rapid release of internal energy.

When integrated with conventional static fracture testing machines, AET enables early identification of failure points by detecting initial damage, mapping the progression of cracks, and offering insights into the underlying mechanisms contributing to material breakdown. Acoustic emission testing allows for monitoring the structural integrity by delivering instant insights into the progression of fractures or damage events. Traditionally, AE is defined as the phenomenon where the rapid release of localized energy within a material generates transient elastic waves, typically occurring in the frequency range of 10–1,000 kHz. During the fracturing of ceramic materials, the frequency of emitted acoustic waves typically ranges between 1 and 500 kHz. Mechanical damages such as cracks and fractures can trigger these traditional AEs.[12,14]

To accurately capture these waves, an energy-harvesting apparatus, such as a transducer or sensor, is required to convert acoustic energy into electrical energy. Acoustic emission testing utilizes these high-frequency sound waves, generated by the sudden release of strain energy within a material during fracture, to detect and measure faults or defects.[14] A variation of AET employs a microphone instead of an ultrasound sensor to capture the initial sounds produced during crack formation in ceramic materials undergoing failure. Once transformed into electrical signals, these sounds enable conventional destructive strength tests to be adapted into non-destructive (ND) techniques.[15]

In an acoustic emission test, the transmission of an electrical signal from a breaking object to a microphone, passing through an amplifier and sent to a switch breaker, is a complex process influenced by various factors that determine the speed of this transmission. One of the factors is the proximity of the microphone to the source, its sensitivity, the quality of the amplifier, and the response time of the switch breaker. Given that the components involved are of high quality and exhibit low latency, it becomes possible to accurately estimate the time taken for the signal to travel.[16] Initially, sound waves pass rapidly through ceramic-like materials and upon reaching an energy harvester-like microphone, are transformed into electrical signals. These signals are then conveyed through cables to the amplifier, which processes them and forwards them to the software analyzer with minimal delay.[17,18]

Integrating a sensitive condenser microphone into FT tests enables precise detection and tracking of noise emissions produced as the sample undergoes failure under load. This system, paired with a customized "cut-off" switch in a UTM, enables the automatic cessation of the loading process when abnormal sounds signaling crack initiation are detected. This approach transforms a conventional destructive FT test into a non destructive (ND) sound harvesting technique significantly improving data accuracy and efficiency.[19]

The aim of this *in vitro* study was to validate a variant of the AET, the super high tension (SHT), for the evaluation of the FT of brittle ceramics in comparison to conventional tests. The null hypothesis suggests that there is no significant difference in the fracture resistance values of the three tested samples when using the SHT method and traditional FT tests.

Materials and Methods

Study Design

The study utilized a comparative experimental design to evaluate the efficacy of SHT in detecting crack initiation and measuring fracture loads in dental ceramics. Two groups of materials (glass and zirconia) were subjected to SHT and standard testing methods. The primary outcome was the fracture load under both test conditions, with a focus on comparing SHT's sensitivity to early crack detection vs conventional methods.

Sample Size Justification

The sample size was calculated based on a power analysis to ensure sufficient statistical power (typically 80%) to detect a significant difference in fracture loads between the two testing methods. Pilot data or previous studies on FT of similar materials were used to estimate effect sizes, leading to a calculation of the number of specimens required in each group to achieve reliable results while minimizing variability and ensuring reproducibility.

Sample Preparation

Zirconia Crowns (Y-TZP)

Fifty monolithic zirconia crowns (MZC) (GC Initial Zirconia Disk® monolithic translucent by GC®) were produced following this protocol:

An artificial mandibular first molar model was prepared by reducing 1.5 mm from the occlusal surface and 1.2 mm from the side walls, finished with a margin design tapering to 0.5 mm, known as a feather edge. Using an extra-oral scanner, computer-aided design (CAD) software, a milling machine, and a sintering furnace, a prepared tooth was scanned, crown designed, milled, and sintered at 1450°C according to the manufacturer's recommendations.

Based on the CAD model, polymethylmethacrylate (PMMA) casts are fabricated via a 3D printing machine. After sandblasting with 50-micron particles at 1.5 bar pressure, the MZC was cemented to printed PMMA models with a universal dual-cure resin cement. The samples were then subsequently light cured for 3 seconds using a curing machine delivering a light intensity of 2500 mW/cm^2. Following 24-hour storage in distilled water at 37 ± 1°C, the samples underwent a thermocycling procedure in agreement with the international organization of standardization, involving 500 cycles alternating between 5 and 55°C, with an immersion time of 20 seconds and a transfer time of 5 seconds.

The MZC were divided into two groups of twenty-five samples: One underwent a conventional load to fracture test and the second a SHT (Fig. 1).

Glass Sheets

Forty similar rectangular glass sheets (1 × 1 cm) were obtained from a 2 mm thick commercial glass piece cut with a laboratory glass cutter, assuring accurate dimensions. The edges of the glass specimens were thoroughly inspected to ensure consistency and surface regularity.

Zirconia Sheets

Forty sheets of non-sintered monolithic translucent zirconia were obtained from a monolithic translucent zirconia block. The block was sectioned into 1 mm thick and 1 × 1 cm slices using a microtome. The sheets were then sintered at 1450°C according to the manufacturer's recommendations[9] and then rigorously examined for any irregularities.

Super High Tension (SHT)

The conventional FT test for the control group involved using a ball-shaped indenter to exert an axial load on the occlusal surfaces of the specimens. The compressive load was applied at a crosshead speed of 0.5 mm/min until the samples were fractured. The load values were recorded with the UTM software. A 2 mm urethane rubber cylinder was positioned between the indenter and the sample to avoid Hertzian damage in both tests (Fig. 2).

The SHT involved positioning a microphone close to the sample inside the UTM. Specifically, the condenser microphone,[20] with a high sensitivity of 20 ± 2 millivolt/Pa, was positioned the closest to the sample. It was connected to an amplifier where it is integrated a motherboard chipset (Fig. 2). To ensure NDT, a custom-designed "cut-off" switch system was included in the UTM setup receiving signal from the amplifier chipset (Fig. 2).

To secure the crown with its PMMA die and prevent noise interference, a preload of 20 N was applied to the sample. Once the desired load was reached, the test recordings were reset, marking the beginning of the evaluation. During the entire process, the amplifier was carefully observed for any unusual changes from the typical machine noise. The chipset was configured to distinguish between standard UTM noise at a rate of 0.5 mm/min and distinct crack sounds generated by the sample. When a crack sound was identified, the chipset would send a command through the cutoff switch to halt the UTM, simultaneously capturing the load measurements in Newtons.

Corrugated foam sheets were used during the tests to minimize external sound interference (Fig. 3).

The UTM software was utilized to gather and save the SHT data, ensuring it was available for detailed future examination.

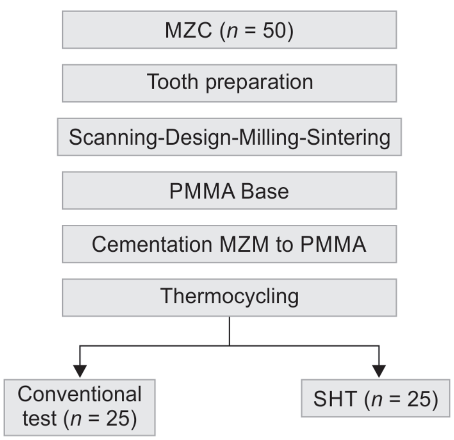


Fig. 1: Flowchart of MZC

Fractography

After completing the static load test, the locations of cracks and fractures within the samples were determined. Subsequently, the tested samples underwent meticulous examination under a low-magnification microscope. Photographic documentation of samples was conducted using a digital single lens reflex (DSLR) camera for further analysis of crack location or non-visible cracks.

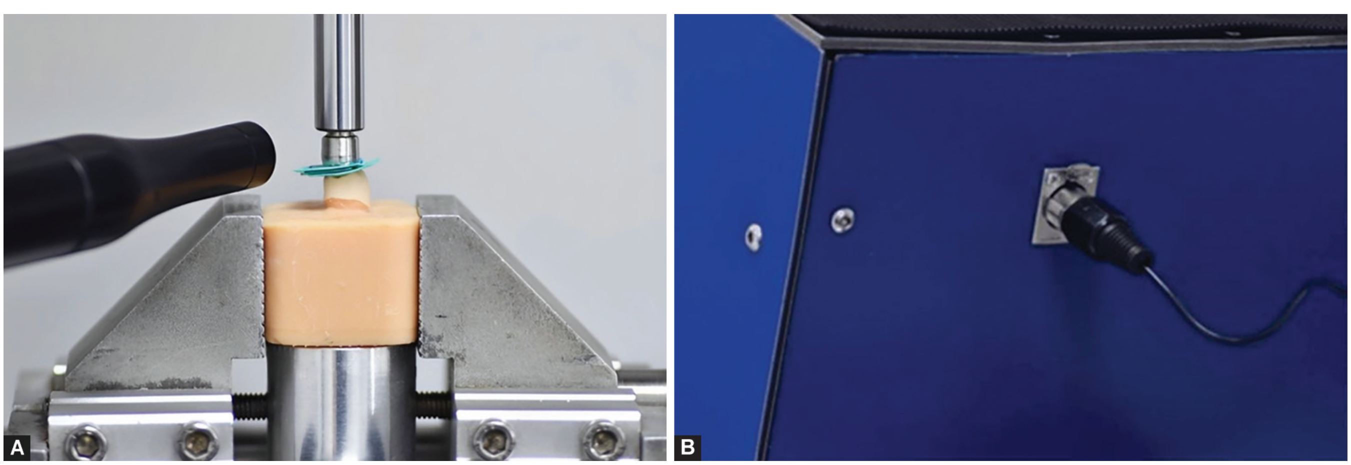
Statistical Analysis

A descriptive analysis was carried out to assess the mean fracture loads across different materials employing diverse techniques. The presentation includes the minimum, maximum, means, and standard deviations (SD) of the fracture loads. The independent samples *t*-test and Mann–Whitney test were applied to identify potential statistically significant differences in the mean fracture load for each material using distinct crack detection techniques. A *p*-value below 0.05 was considered indicative of statistical significance. The statistical analysis was executed using IBM SPSS Statistics 25 software.

Results

The study's final sample comprised 130 items, divided into three groups for fracture strength testing using different crack detection methods. The groups included 40 glass sheets, with 20 subjected to the SHT and the remaining 20 to conventional methods. Another 40 zirconia sheets were tested, equally split between SHT and conventional techniques. Lastly, 50 zirconia crowns were assessed, with half evaluated using SHT and the other half using traditional testing methods, to compare the efficacy of the SHT against conventional approaches in detecting fractures.

The results were consolidated in Table 1, showing fracture loads across various materials using SHT and conventional techniques:



Figs 2A and B: (A) Custom test setup with zirconia crown on PMMA die; (B) Cut off switch in the UTM

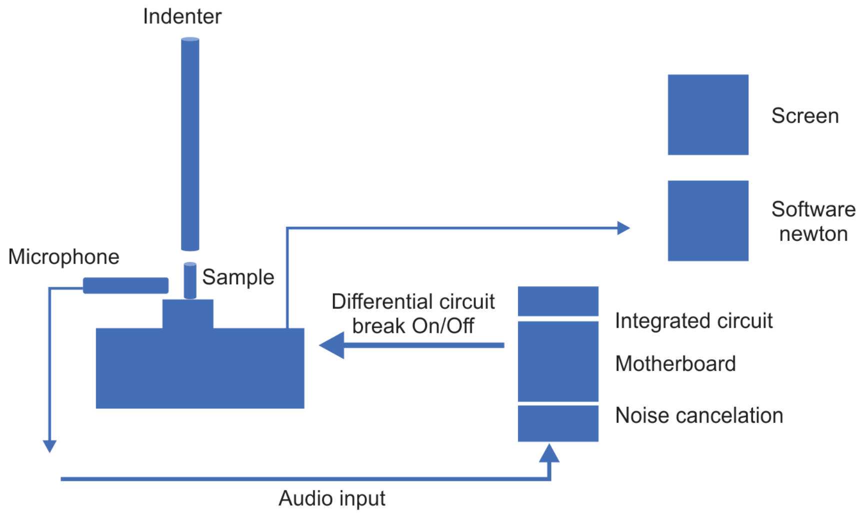


Fig. 3: Sound harvesting test setup

Table 1: Fracture load (N) of different materials using two techniques

	SHT				*Conventional*				
	Min	*Max*	*Mean*	*SD*	*Min*	*Max*	*Mean*	*SD*	*p-value*
Glass sheets	338.00	803.00	650.46	110.38	205.00	945.90	691.41	155.92	0.010[a]
Zirconia sheets	80.00	111.00	95.25	7.78	76.00	238.00	112.75	31.26	<0.001[a]
Zirconia crowns	217.99	1748.00	1108.99	327.89	840.00	1840.00	1292.52	271.42	0.036[b]

[a]Mann–Whitney test; [b]independent samples' *t*-test. SHT, super high tension

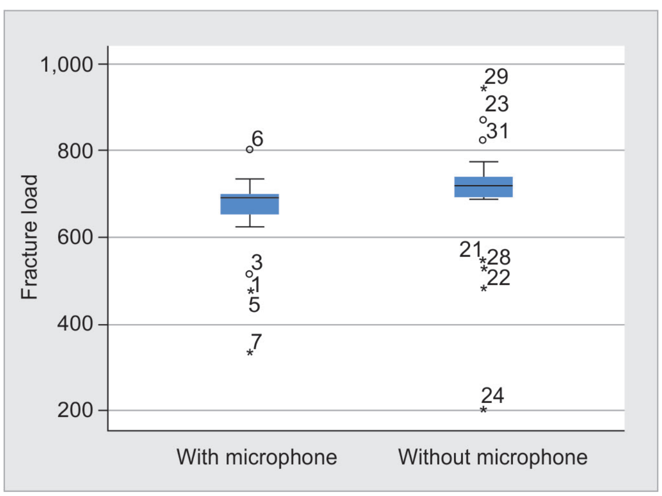


Fig. 4: Fracture load (N) of different glass sheets using two techniques ($n = 40$)

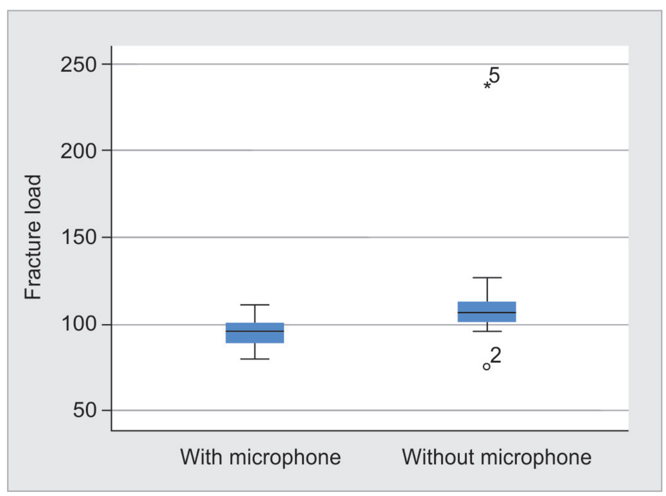


Fig. 5: Fracture load (N) of different zirconia sheets using two techniques ($n = 40$)

Glass Sheets

For the SHT group, the fracture load ranged from 338 to 803 N, with a mean of 650.46 N and an SD of 110.38. For the conventional testing group, the load ranged from 205 to 945.90 N, with a mean of 691.41 N and an SD of 155.92. The *p*-value indicating statistical significance between these groups was 0.010, demonstrating a significant difference (Fig. 4).

Zirconia Sheets

In the SHT group, the fracture load ranged from 80 to 111 N, with a mean of 95.25 N and an SD of 7.78. For the conventional testing group, the load ranged from 76 to 238 N, with a mean of 112.75 N and an SD of 31.26. The difference between these methods was highly significant, with a *p*-value of less than 0.001 (Fig. 5).

Zirconia Crowns

For the SHT group, the fracture load ranged from 217.99 to 1748 N, with a mean of 1108.99 N and an SD of 327.89. For the conventional testing group, the load ranged from 840 to 1840 N, with a mean of 1292.52 N and an SD of 271.42. The statistical significance of the difference between these groups was indicated by a *p*-value of 0.036 (Fig. 6).

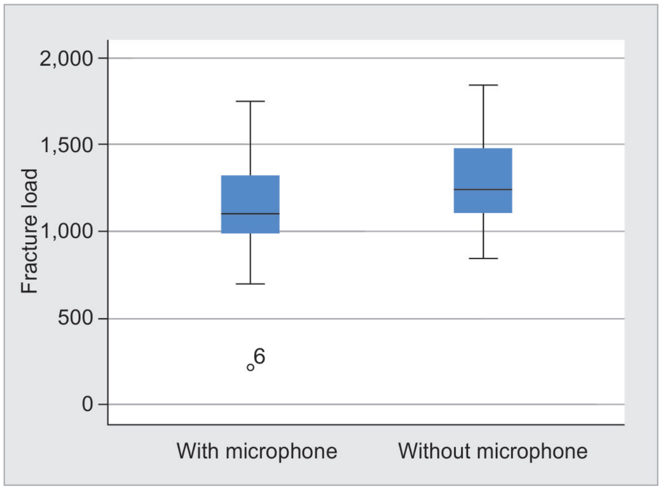


Fig. 6: Fracture load (N) of different zirconia crowns using two techniques ($n = 50$)

The results show significant differences in fracture load values between SHT and conventional testing methods across all material types. Glass sheets tested with SHT exhibited a lower mean fracture load (650.46 N) compared to conventional testing (691.41 N). Zirconia sheets tested with SHT also showed a lower mean fracture load (95.25 N) compared to conventional methods (112.75 N). Similarly, zirconia crowns tested with SHT had a lower mean fracture load (1108.99 N) compared to conventional testing (1292.52 N). These findings highlight the efficacy of the SHT in detecting fractures at lower loads, thereby enhancing the accuracy of fracture load measurements.

Discussion

The study explored the efficacy of the modified SHT method for evaluating the fracture resistance of brittle ceramic materials, compared to standard testing techniques for validation. Our findings demonstrated significant disparities in fracture loads when employing SHT in comparison to standard test.

Glass sheets assessed with SHT exhibited a mean fracture load of 650.46 N, while standard testing yielded a higher average of 691.41 N. Zirconia sheets under SHT had an average fracture load of 95.25 N, in comparison to 112.75 N achieved without this technique. In a similar trend, zirconia crowns with 0.5 mm thick margins registered an average load of 1108.99 N with SHT, which was less than the 1292.52 N obtained through standard testing.

These observed differences were not only consistent but also statistically significant. The p-values obtained <0.001 for zirconia sheets, 0.036 for zirconia crowns, and 0.010 for glass sheets confirmed the significance of the findings.

Furthermore, we noted that glass sheets subjected to SHT had fracture loads ranging from a minimum of 338 to a maximum of 803 N. Without SHT, these sheets displayed a wider load range from 205 to 945.90 N. For 0.1 mm thick zirconia sheets, SHT captured a narrower load spectrum of 80 to 111 N, whereas non-SHT methods disclosed a broader range from 76 to 238 N. Zirconia crowns tested with SHT tolerated loads from 217.99 to 1748 N, significantly narrower than the 840 to 1840 N range identified without SHT.

Failure load values from the SHT were lower than those from conventional methods for glass and zirconia sheets and crowns, due to SHT's ability to detect cracks before catastrophic failure. This results in more precise load-bearing values. Outliers in fracture load distribution are likely due to material impurities. Sound harvesting test's unique detection of early crack initiation and propagation leads to lower recorded fracture loads, making it more reliable for early detection and preventive measures. While conventional methods measure maximum failure load, potentially missing early fracture signals, SHT provides detailed and accurate fracture resistance assessments, offering a valuable ND evaluation tool.[20]

The mean fracture load results differences obtained using the two techniques align with findings from prior research on NDT. For example, Ellakwa et al. assessing the impact of various detection techniques on crown fracture load, observed that the fracture load was notably influenced by the detection method.[21]

Careful consideration was given to material selection to optimize sound isolation, transmission, and collection, which are critical factors in the effectiveness of the SHT. For the die material, we preferred those that resemble the mechanical properties of natural teeth. The use of PMMA as a die material in this study was justified based on its low sound transmission properties. Nakamura et al.'s research demonstrated that resin-based dies have a significantly lower elasticity modulus compared to zirconia crowns. Additionally, PMMA, a material frequently used for temporary dental restorations, has been studied for its acoustic properties. Chen et al. reported that PMMA resin exhibits a modulus of elasticity around 2100.05 ± 114.28 MPa.[22] Consequently, employing PMMA and zirconia crown specimens for fracture tests can yield results with significant clinical relevance.

The brittle materials used in the study were zirconia and glass. They both have similar properties. Their inherent brittleness and sound transmission capabilities make them suitable materials for SHT. Furthermore, zirconia crowns are extensively utilized in fixed partial dentures, both as crowns and bridge materials. Zirconia was chosen for SHT validation because of its favorable acoustic characteristics, making it ideal for analyzing crack initiation and propagation in fragile dental ceramics. Moreover, its superior flexural strength promotes efficient transmission and movement of acoustic waves.[23,24]

Glass was chosen as an additional material for its distinctive sonic properties. Known for its variable hardness, depending on its composition, glass has a relatively low tensile strength, predisposing it to tensile fractures, yet it maintains robust compressive strength. Glass's lower elastic modulus means it has a limited capacity to deform elastically under stress, making it more likely to break. When it does fracture, glass transmits sound waves that can be captured during acoustic testing. This was particularly considered with our 2 mm thick glass sheet samples, facilitating a comparative study alongside zirconia material in our investigation.[25]

This study optimized components in the sound transmission process to harness electrical energy from mechanical vibrations efficiently. An acoustic signal from a crack in glass material, captured by high-quality microphones, travels through the glass at up to 5,500 m/s, far outpacing sound in the air. Upon reaching the microphone, the energy is converted to an electrical signal, rapidly transmitted through cables to an amplifier and switch breaker at near-light speeds. The frequency of acoustic waves during dental ceramic fracture typically falls within 1–500 kHz, captured by a high-sensitivity, pre-polarized condenser microphone. The speed of this transition depends on minimal processing time in the amplifier, the cables' electrical properties, and material characteristics such as composition and impurities. In the FT test, a strategically positioned high-sensitivity microphone records noise emissions during sample loading, enhancing the detection of crack initiation. To minimize external sound interference, corrugated foam sheets and a preliminary load of 20 N were used, along with a urethane rubber cylinder to prevent Hertzian damage. This setup, with the microphone channeling sound waves through electric external line return (XLR) cables to the amplifier, is crucial for accurate technique execution, impacting the transformation speed of the raw wave through the chipset to the cut-off switch.[26,27]

Amplifiers significantly impact signal travel time estimation and latency through various design and setting parameters. Techniques like digital pre-distortion mitigate distortion and ensure linearity while timing adjustment units optimize output power by adjusting the delay between input signals and power supply voltages. Additionally, noise and signal power measurements assess amplifier performance, directly influencing signal quality and latency in communication systems. Cable length, quality, and electrical characteristics also affect signal velocity, with modern amplifiers typically maintaining low latency. The swift electrical transmission, compared to slower sound speeds, highlights differences in crack propagation rates in materials. Factors such as temperature, humidity, and material defects can affect sound transmission speed, emphasizing the need for material-specific considerations in acoustic testing. The speed of sound in glass and Zirconia materials is high, and once cracked, the resultant electrical signal travels quickly through cables, with amplifiers processing and transmitting the signal efficiently, assuming low-latency amplifiers.[28]

Acoustic emission testing has been previously used to detect the fracture of different dental structures, but few studies have used it for evaluating the load-bearing capacities of dental ceramics. Ereifej et al. used the AET method to detect the onset of fractures in ceramic crowns,[29] while Vallittu applied it to study the fracture pattern in composite veneers reinforced with woven glass fibers.[30] Similarly, Kim and Okuno explored the micro-fracture behavior of composite resins containing fillers with irregular shapes using this technique.[31]

The application of sound harvesting tests in the field of dental ceramics is still under-researched and not widely understood. Our study aligns with earlier research, highlighting that the chosen approach for crack detection plays a crucial role in altering the fracture load values recorded in different materials, potentially affecting the accuracy of mechanical evaluations and the interpretation of material strength.

Gdoutos and Konsta–Gdoutos compared different crack detection methods and found that acoustic testing showed higher sensitivity and accuracy in detecting cracks compared to visual inspection and dye penetration testing. This finding is consistent with the results of our study, where statistically significant differences were observed in the mean fracture load using the different crack detection techniques, particularly for glass sheets, 0.1 mm zirconia sheets, and zirconia crowns.[32]

Another study conducted by Wang et al. showed that acoustic testing can detect cracks and defects in dental materials with better accuracy.[33,34] Zhang et al. investigated the effect of different crack detection techniques on the fracture resistance of glass ceramic crowns and reported that using a microphone during crack detection led to a decrease in the fracture load.[35] Thus, this shows that the result of our study supports the findings of studies found in the literature. Nonetheless, the presence of outliers in the distribution of fracture loads of glass sheets using both techniques could be attributed to the impurities present in the material, considering its commercial nature.

Finally, the results of our study lead us to reject the null hypothesis, suggesting that there is indeed a significant difference in the load values between the SHT and conventional FT test. Its ability to detect early cracks enhances fracture resistance assessments, improving the durability and prognosis of dental restorations.

However, the study acknowledges limitations in the variability of tested samples, suggesting that future clinical applications should include a wider range of materials to improve the prognostic capabilities and the development of more durable ceramic restorations.

Future research involving a broader range of dental materials could enhance the accuracy, generalizability, and clinical implications of these findings, particularly for applications in posterior fixed partial dentures and dental implants.

Conclusion

The study demonstrates that the SHT offers notable improvements over traditional methods in identifying crack initiation in brittle ceramics under stress, allowing for more accurate assessments of fracture behavior. Compared to standard testing, results showed that SHT yielded lower fracture loads for glass sheets and zirconia materials, highlighting its advantage in detecting cracks at lower forces. These findings suggest that SHT provides a precise and reliable method for evaluating the fracture resistance and toughness of dental ceramics, which could enhance the accuracy of future material assessments.

Author's Contributions

Conceptualization: Camille Haddad Methodology: Camille Haddad, Jean Gebran Formal analysis: Camille Haddad Writing – original draft preparation: Camille Haddad Writing – review and editing: Camille Haddad Supervision: Amine Zoghbi.

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