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Assessing Fracture Resistance of Dental Ceramics with a Sound Harvesting Acoustic Emission Test

Camille Haddad*, Jean Gebran, Amine Zoghbi

Department of Prosthodontics, Saint Joseph University, Beirut, Lebanon

Research Article

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***For Correspondence:** Camille Haddad, Department of Prosthodontics, Saint Joseph University, Beirut, Lebanon

Email: ball.ellen@gmail.com

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ABSTRACT

Introduction: Evaluating the fracture resistance of dental ceramics such as monolithic zirconia crowns is crucial for assessing their durability. Conventional destructive laboratory tests often fail to accurately evaluate the timing and failure crack formation of brittle materials. Non-destructive testing methods, such as acoustic emission testing, offer an alternative by providing valuable data on the material properties that cause damage to the samples. This *in vitro* study aimed to evaluate the sensitivity of a sound harvesting modified acoustic emission test by comparing the fracture resistance of posterior Monolithic Zirconia Crowns (MZCs) measured *via* a test with that of a conventional fracture toughness test.

Materials and methods: A modified acoustic emission test, the sound harvesting setup, featuring a microphone, custom audio chipset, and cut-off switch integrated into a universal testing machine, was compared to a conventional fracture toughness test to measure fracture loads on 50 posterior monolithic zirconia crowns divided into two groups.

Results: The sound harvesting test recorded a mean fracture load of 1108.99 N, which was significantly lower than that 1292.52 N measured using the conventional test, indicating a more sensitive detection of fractures.

Conclusion: The sound-harvesting test shows potential as an alternative testing method for dental ceramics, highlighting its ability to identify failures at lower loads.

Keywords: Monolithic zirconia; Fracture resistance test; Acoustic testing; Sound harvesting test; Brittle ceramics

INTRODUCTION

Over the past decades, ceramic materials have gained significant importance in dentistry due to their optical properties and biocompatibility, making them ideal for dental prostheses notably, monolithic zirconia material commonly used in posterior crowns owing to their high strength and aesthetic properties, which contribute to the stability and longevity of posterior Fixed Partial Dentures (FPDs) [1].

However, ceramics are naturally brittle with limited tensile strength and susceptibility to time-dependent stress failure when subjected to various forces within the oral cavity, including masticatory forces, occlusal stresses and thermal changes. As a result, fracture resistance is a critical aspect of their performance as it directly affects the longevity and durability of dental restorations [2,3]. *In vitro* studies are crucial for evaluating the potential clinical performance of new dental materials before embarking on costly and time-consuming clinical investigations. Assessing their fracture resistance allows clinicians and researchers to evaluate their suitability for different clinical applications and predict their clinical performance over time. This evaluation process involves testing materials under simulated loading conditions to measure their resistance to crack propagation and failure [4].

Several Destructive Testing (DT) techniques, including strength and Fracture toughness Tests (FT), have been used to evaluate the performance and properties of dental ceramics. Using a universal testing machine with a spherical stainless-steel indenter, the FT test is based on applying a compressive load to the occlusal surface of the samples at a speed of 0.5 mm/min, to measure the force required to cause fracture. However, DT, although effective, has several limitations. First, these tests involve sample destruction, rendering the samples unusable for further testing, which can be expensive for materials that are expensive or available in limited quantities. Second, DT provides only a single-point evaluation of the material's properties at a specific moment, failing to capture early changes, such as crack initiation, over time owing to factors such as aging [5,6]. Finally, DT offers only a limited assessment of failure modes, focusing primarily on determining the maximum failure strength, without providing detailed insights into specific failure mechanisms. To address these limitations, Non-Destructive Testing (NDT) methods offer an alternative approach for evaluating brittle dental ceramics, preserving their functionality while assessing structural integrity, quality and performance [7].

NDT techniques are valuable tools for assessing the integrity of structures and materials in various industries including dentistry and ceramic production. These methods enable researchers to evaluate materials or components without causing damage, offer significant cost savings, and ensure the quality of engineered systems and products. In ceramic production, NDT methods are crucial for detecting structural defects without causing damage. However, certain key areas still lack viable testing methods, emphasizing the need for the development of additional accurate and effective NDT techniques to ensure ceramic product quality [8]. The commonly used NDT methods for ceramic materials include visual inspection, penetration testing, ultrasonic testing, radiographic testing, infrared thermography, laser ultrasonics, X-rays, optical coherence tomography, laser ultrasonics, computed tomography and Acoustic Emission Testing (AET) [9].

The fundamental principle of AET involves harvesting electrical energy from mechanical vibrations generated by sound waves effectively identifying the initiation and progression of failure in brittle materials while maintaining the integrity of the tested samples. It is used to assess properties such as the fracture strength. Integrated with conventional static fracture test machines, AET can determine the onset of failure, locate the initial damage site, track damage propagation and expose the complex mechanisms leading to material failure [10,11].

One variant of AET involves using a microphone instead of an ultrasound sensor to harvest the early sound emitted by crack formation in the failing ceramic material. When converted to electrical signals, the emitted noise can be used to transform traditional destructive strength testing into NDT by activating a cutoff switch to halt the universal testing machine UTM load during crack initiation [12].

The process of transmitting an electrical signal from a material such as zirconia to a microphone, amplifier, or switch breaker involves various factors that influence the speed of transmission. These factors include the distance between the material and microphone, microphone sensitivity, amplifier quality and switch breaker response time. The travel time of the signal can be estimated by assuming that high-quality elements with low latency are used. Sound waves travel quickly through materials such as ceramics, but once they reach the microphone, they are converted into electrical signals transmitted through a cable to the amplifier. Ideally, the amplifier processes the signal and sends it to the switch breaker with minimal latency.

The speed of the signal in the cable is influenced by the cable length, quality and electrical properties such as resistance and capacitance. In addition, the processing time of the amplifier depends on its design and settings, with modern amplifiers typically having low latency. Because they travel at the speed of light, the electric signal transmission through wires is much faster than the speed of sound in air. Material characteristics, such as composition and stress conditions, affect the crack propagation speed. The ability of the electrical circuit to rapidly stop the test upon crack initiation is vital for accurate data collection. By integrating a high-sensitivity microphone within the fracture toughness test, we could precisely monitor the noise emissions generated by the samples during loading. The positioning of the microphone just 1 cm away from the sample ensured optimal sensitivity for detecting even subtle crack sounds. This setup, coupled with a custom-designed “cut-off” switch system, enables the automatic halting of the load process upon detecting abnormal sounds indicative of crack initiation.

The objective of this *in vitro* study was to evaluate the sensitivity of a modified acoustic emission test by comparing the fracture resistance of posterior Monolithic Zirconia Crowns (MZCs) measured *via* a test with that of a conventional fracture toughness test.

MATERIALS AND METHODS

Sample preparation

Fifty zirconia monolithic crowns (Initial Zirconia Disk® monolithic translucent produced by GC®, Leuven, Belgium) were tested in this experiment. They were evenly distributed into two groups: 25 crowns (group 1) underwent SHT, while the remaining 25 served as a control group with FT (group 2). This study employed a blinded assessment for unbiased evaluation.

An operator milled a single intact artificial mandibular first molar (Frasaco®, Tettnang, Germany) using a turbine handpiece and diamond burs of different diameters. The occlusal surface area (functional cusps) was reduced by 1.5 mm (functional cusps). The axial walls were tapered by four degrees and reduced by 1.2 mm (Komet® 8862; Lemgo, Germany). The artificial tooth was prepared with a feather-edge margin of 0.5 mm [13]. All edges were rounded and polished with a handpiece micromotor, silicone polishing burs of different grain diameters and a polishing brush with polishing paste (Dialux® Blanc, Salisbury, UK). The tooth that had been prepared was subsequently placed horizontally along the axis of the customized metallic mold, which held cold-cure acrylic resin with dimensions measuring 2.5 × 2.5 × 3 mm^3.

The die, including an artificially prepared tooth and an acrylic base, was scanned using a laboratory scanner (Dental Wings, Exocad, 3 Shape®, Montreal, CA, USA). The Standard Tesselation Language (STL) file was analyzed using Computer-Aided Design (CAD) software (Mayka Dental V6, Picasoft®, Yangon, Burma).

The 3D virtual die was adjusted in the CAD software according to the manufacturer’s directives with a 0.5 mm feather-edge margin. A space of 40 µm dedicated to the cement was formed using the gap thickness tool in CAD software. Using the CAD die, 50 Polymethylmethacrylate (PMMA) dies (Figure 1) were printed (Formlabs 2®, Somerville, MA, USA) using a 3D printing

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machine (Yenadent®, Istanbul, Turkey).

The MZCs were milled using a five-axis milling unit (Kavo Everest®, Charlotte, NC, USA) from four monolithic zirconia discs measuring 98 × 16 mm (Initial Zirconia Disk® monolithic translucent by GC®). The MZCs were sintered at 1500°C in a sintering furnace according to the manufacturer's instructions.

The MZCs were cemented onto the PMMA dies with resin cement (G Cem one®, GC®) by the same operator after sandblasting (50 μm 1.5 bar) [14]. A 1 mm rubber cylinder was made and placed between the indenter tool and crown to prevent direct damage and evenly distribute the forces. To secure the sample on the die, a universal testing machine applied a 20 Newton vertical load on the top surface. The same operator repeated this process for each MZC-PMMA die cementation. All samples were kept in an incubator for seven days prior to mechanical testing.

Figure 1. Monolithic zirconia crown on PMMA base.

Subsequently, a thermocycling procedure was performed. It consisted of 500 cycles alternating between 5°C and 55°C. The immersion time in each bath was 20 s and the transfer time was 5 s [15].

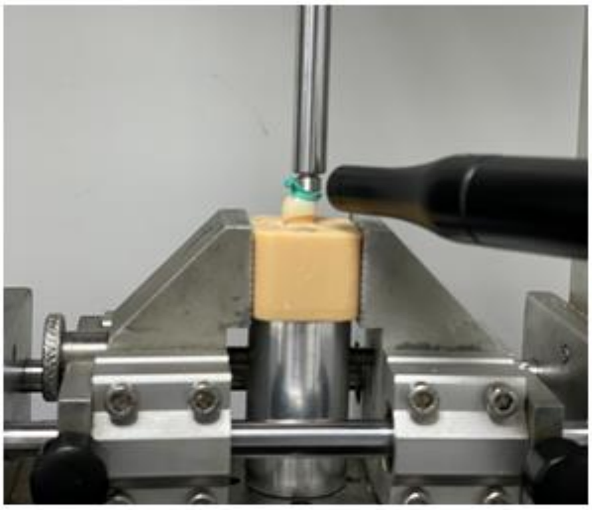
By integrating a high-sensitivity microphone into the fracture toughness test, we were able to precisely monitor the noise emissions generated by the samples during loading. The positioning of the microphone just 1 cm away from the sample ensured optimal sensitivity for detecting even subtle crack sounds. This setup, coupled with a custom-designed "cut-off" switch system, enables the automatic halting of the load process upon detecting abnormal sounds indicative of crack initiation.

Fracture toughness tests

To prevent Hertzian damage during both tests, a 2 mm urethane rubber cylinder was placed between the indenter and sample. To perform the conventional fracture toughness test, a ball-shaped indenter was used to create an axial load on the occlusal surfaces of the samples. A compressive load was applied at a crosshead speed of 0.5 mm per minute until fracture. The load values were recorded using UTM [16].

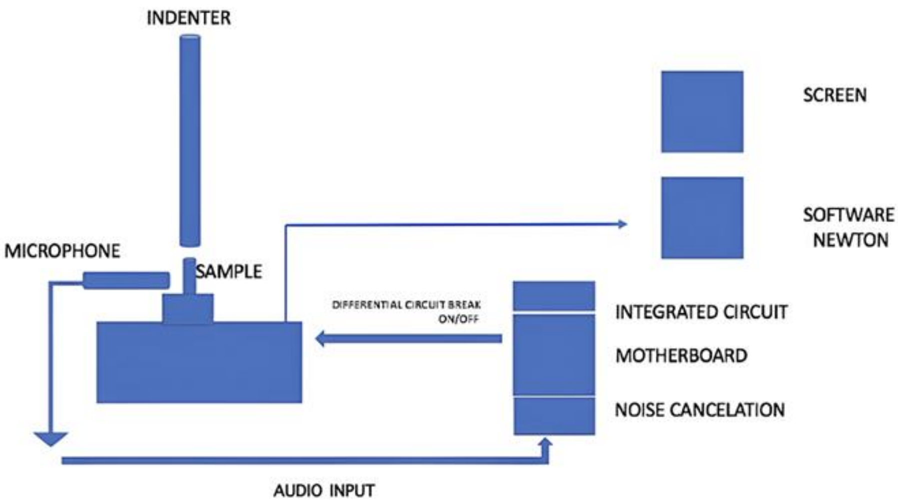
The sound Harvesting test was performed by placing a microphone near the sample within a Universal Testing Machine (UTM) (YLE® GmbH, Waldstraße Bad König, Germany). Specifically, a MiniSPL® microphone produced by NTI was positioned 1 cm from the sample. This microphone, with a high sensitivity of 20 ± 2 millivolt/Pa, was connected to an amplifier (Avalon Design 737®, Nashville, TN, USA) integrated into a motherboard chipset. To ensure non-destructive testing, a custom-designed "cut-off" switch system was included in the UTM setup (Figure 2).

Figure 2. Sound harvesting setup and the UTM machine.



To consolidate the crown with its PMMA die and prevent noise interference, a preload of 20 Newton was applied to the sample. After reaching this desired load, the recordings were reinitialized and the desired test was started. Throughout the test, the amplifier was continuously monitored for any deviations from normal machine noise. The chipset was programmed to differentiate between normal UTM noise descending at a speed of 0.5 mm/Mn and crack sounds emitted by the sample. Upon detecting a crack sound, the chipset triggered an electric command through the cutoff switch to halt the UTM, automatically recording the load values in Newtons (Figure 3). To minimize external sound interference, corrugated foam sheets from Cactus® USA, were used for noise cancellation during the tests.

Figure 3. The sound harvesting test setup.



Visual inspection of cracks

Following the static load test, cracks in the samples were identified (Figure 4) and a subsequent meticulous examination was performed under a low-magnification microscope (Leica Microsystems®, Wetzlar, Germany). Photographic documentation of the samples was performed using a DSL camera (Nikon®, Tokyo, Japan) for further analysis of the crack location or potential fractures. Photographs of all samples were taken at various positions and analyzed to determine the location of the crack or eventual fracture (Figure 4).

Figure 4. Crack on occlusal side of monolithic zirconia crown.

Statistical analysis

Statistical analyses were performed using SPSS Statistics for Windows (version 25.0; IBM® Corp, Armonk, NY, USA). An independent samples t-testing was conducted to examine whether there was a significant difference in the load means between the two groups (group 1 and group 2). In every situation, a p-value less than 0.05 was considered statistically significant.

RESULTS

Table 1 presents the fracture loads (in Newtons, N) for the MZCs obtained using the SHT and conventional techniques. With SHT, the fracture loads ranged from 217.99 N to 1748.00 N, with a mean of 1108.99 N and Standard Deviation (SD) of 327.89. Without SHT, the fracture loads ranged from 840.00 N to 1840.00 N, with a mean of 1292.52 N and SD of 271.42. Statistical analysis revealed a significant difference (p=0.036) between the two techniques, as determined by the independent sample t-test.

Table 1. Fracture loads in Newtons (N) obtained for the 2 testing methods (N=50).

	SHT				**Conventional**				**p-value**
MZC	**Min**	**Max**	**Mean**	**SD**	**Min**	**Max**	**Mean**	**SD**	
	217.99	1748	1108.99	327.89	840	1840	1292.52	271.42	0.036

Classification of crack/fracture

Table 2 provides a comparison between the SHT and the conventional methods for detecting fractures and cracks.

The sound harvesting test detected six out of 25 fractures (24%), while the conventional method detected 18 out of 25 fractures (72%), resulting in a total of 24 fractures out of 50 samples (48%). For cracks, SHT detected 19 out of 25 cases (76%), whereas in the conventional method, only 2 samples out of 25 cracked (28%).

Table 2. Comparison between the Sound Harvesting Test (SHT) and the conventional test for detecting fractures and cracks. (n=50).

	SHT	Conventional	Total
	n (%)	n (%)	n
Fracture	6 (24%)	18 (72%)	24 (48%)
Crack	19 (76%)	7 (28%)	26 (52%)
Total	25 (50%)	25 (50%)	50 (100%)

DISCUSSION

In the present study, the effectiveness of a sound harvesting test was examined for its ability to detect cracks in brittle dental ceramics such as monolithic zirconia crowns. The performance of SHT in monitoring fracture events was evaluated and compared with a conventional fracture toughness test that measures fracture loads in Newtons (N).

The results revealed that SHT identified lower mean fracture loads of 1108.99 N compared to 1292.52 N with standard tests, showcasing SHT's higher sensitivity of SHT in detecting fractures at earlier stages. Statistical analysis showed that these differences were significant, supporting the hypothesis that the SHT can provide more accurate fracture load assessments.

Statistical analyses supported by significant p-values emphasize the non-random nature of these differences, consistent with prior research, which emphasized the influence of detection methods on crown fracture load [17].

For cracks, SHT was detected (76%), whereas in the conventional method, only two out of 25 samples cracked (28%). Overall, the SHT exhibited a higher sensitivity for detecting cracks.

Regarding the selection of research materials, careful consideration was given to ensuring optimal sound isolation, transmission and collection. The die material was chosen to replicate the characteristics of the natural teeth. PMMA resin was chosen because of its similarity to natural teeth in terms of modulus of elasticity and acoustic response [18].

Nakamura et al., revealed that the modulus of elasticity of a resin-based die is lower than that of zirconia crowns. In addition, previous studies have investigated the acoustic response of PMMA. Chen et al., found that PMMA resin had an approximate modulus of elasticity of 2100.05 ± 114.28 MPa. Chen et al., found that PMMA resin has an approximate modulus of elasticity of 2100.05 ± 114.28 MPa.

Thus, using PMMA/MZC specimens in load-to-fracture tests can yield clinically relevant results.

Zirconia, a widely used material in dentistry, was chosen as the material for validating SHT owing to its technical acoustic properties, which makes it suitable for studying crack initiation and propagation in dental ceramics, and because of its high flexural strength, enabling effective transmission and propagation of acoustic waves [19,20].

Acoustic Emission Testing (AET) is a non-destructive method used in various industries, including dentistry, to detect and analyze stress waves resulting from sudden stress redistribution in materials. AET relies on harvesting energy released from the object under examination. Equipped with specialized tools, such as sensors, amplifiers, and filters, it collects failing energy data from the tested samples.

Roques et al., applied Acoustic Emission (AE) testing to analyze bone cement failure through fatigue tests, suggesting AE's usefulness as a preclinical measurement of the strength of cemented implants. Silva et al., found that the precision of acoustic testing was comparable with that of micro-CT for detecting cracks in dental ceramics, demonstrating its reliability. Moreover, Lim et al., studied microcrack growth in ceramic/dentin interfaces, revealing AET's promise in assessing the

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integrity of such materials. These studies collectively affirmed the efficacy of AE methods in detecting material defects, which is crucial for ensuring the longevity of dental restorations. However, owing to the complexity of dental restorations, adapting ultrasonic receptors to the test setup can be challenging.

Our sound harvesting test applies Acoustic Emission Testing (AET) to dental materials by converting sound-generated vibrations into electrical energy to evaluate material strength and fracture resistance.

The incorporation of an amplifier and motherboard chipset further augmented the sensitivity and accuracy of the SHT setup. By programming the chipset to differentiate between normal machine noise and crack sounds, we ensured the reliable detection of crack events during testing. Moreover, the automatic recording of load values in Newtons upon the detection of crack sounds enables efficient data collection and analysis. The use of noise-cancellation materials, such as corrugated foam sheets, further minimizes external sound interference, thereby controlling the environmental variables.

While SHT is generally nondestructive, a minor fraction of the samples catastrophically broke during testing. This could be due to the transmission pathway of the signal from a zirconia crack to a microphone and then through an amplifier to a switch breaker, which involves several variables, such as distance, microphone sensitivity, electric wires (XLR (Pig Hog PHM10 8 mm®)), amplifier quality (93 dB) and the speed at which the switch breaker reacts as well as the material characteristics, including composition, impurities, stress conditions and specific wave types involved in crack propagation. In addition, sound travels through air at speeds influenced by temperature, humidity and pressure; under standard conditions, it is approximately 343 m/s in dry air, which is much slower than the electrical signals in wires. These electrical signals can reach nearly the speed of light.

In the field of dental ceramics, the application of sound-harvesting tests has been relatively unexplored. Our research supports previous findings indicating that the crack detection method significantly impacts the measured fracture loads in various materials. Wang et al.,'s work, which showed the superior sensitivity and specificity of acoustic methods over dye penetration for finding cracks in dental ceramics, supports our observations of reduced fracture loads when using sound-based detection. Furthermore, Al-Zubaidi et al., and studies by Zhang et al., and Wang et al., reinforce this perspective, reporting that acoustic testing, particularly with the use of a microphone, is more sensitive than conventional testing.

Complementing these acoustic methods, Akono et al., introduced a micro-scratch technique using scratch data for the quantitative assessment of material toughness, offering a highly reproducible and minimally invasive alternative to acoustic and optical assessments. Akgün et al., used impulse noise testing to detect defects in ceramic materials, adding another layer to the evolving testing designs. These studies suggest that variations in the testing results are influenced by the choice of method, material types, and specifics of the detection techniques employed, contributing to a broader understanding of how dental materials respond to different testing modalities. Nevertheless, our study contributes to the current understanding by examining how brittle dental materials react acoustically. However, this study was limited to a single sample selection. Future studies should include more brittle materials.

CONCLUSION

In conclusion, this research suggests that the sound harvesting test provides benefits compared with conventional fracture toughness tests for identifying the timing of early crack formation in brittle materials subjected to stress, thereby allowing for a more accurate evaluation of the fracture toughness of dental ceramics. However, further research on a wider range of brittle materials is warranted to support their broader application in dentistry.

AUTHOR CONTRIBUTIONS

Conceptualization, C.H.; methodology, C.H. and J.G.; formal analysis, C.H.; writing—original draft preparation, C.H.; writing-review and editing, C. H.; supervision, A.Z. All authors have read and agreed to the published version of the manuscript.

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INSTITUTIONAL REVIEW BOARD STATEMENT

No human participants or live animals were included in this study; therefore, ethical considerations were not applicable. This research adhered to the guidelines and ethical standards through strict study methodology, proper data management and accurate documentation of findings.

INFORMED CONSENT STATEMENT

Not applicable.

DATA AVAILABILITY STATEMENT

Not applicable.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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