Dentistry 3000

Vol 14, No 3 (2026)
ISSN 2167-8677 (online)
DOI 10.5195/d3000/2026.1654

Superior Stabilized Zirconium Compacts Preparation with Minimum Thickness That Is Suitable for Fabrication of Dental Crowns and Bridges

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Abstract

The stabilized zirconia, particularly 3Y-TZP is used for fabrication of dental crowns and bridges through supply of it as compacts suitable to be shaped by CAD/CAM machines and then sintering via special sintering program provided the company source. Among the best celebrated product of this type is that of GC company that is found in the local and global market. Although the sintered stabilized zirconia has high mechanical properties; the design of the dental crowns and bridges has complex shapes and thin edges that weaken the mechanical properties. For that reason, the dentist still experiences the chipping or the breakup of the thin edges because of accumulated stresses during mastication, when occasionally dropped, or during cementation by the dentist. Accordingly, the need is raised for the development of the stabilized Zirconia to improve its mechanical properties. In this work, the compressive strength of the stabilized Zirconia has been improved noticeably and strain at maximum load was also improved, which give a lesser opportunity for chipping and breakup that experienced by the clinicians especially at thin edges. The properties have been achieved via adding of the low-cost boron oxide although the utilized zirconia powder was not in the nanoscale and high-cost additions was not used, also, the traditional compaction technique has been used. The prepared materials were characterized via measurements of density, crystalline phases via XRD, compressive strength, the strain at maximum load, the microstructure at the fracture surface, in addition to the surface morphology via SEM.

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Citation: Al-Ameedee WHM et al. (2026) Superior Stabilized Zirconium Compacts Prepartion with Minimum Thickness That Is Suitable for Fabrication of Dental Crowns and Bridges. Dentistry 3000. 1:a001 doi:10.5195/d3000.2026.1654
Received: October 13, 2026
Accepted: October 20, 2026
Published: October 22, 2026
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Introduction

In dental prosthesis, ceramics could be classified according to the type of used ceramic and method of manufacture. Two kinds of prosthesis: First is the one that rely on a metal core, and a ceramic shell consists of porcelain ceramic (mainly leucite phase) sintered on the metal core. The second type is the newest one, which consists of a durable ceramic core with a porcelain ceramic shell sintered on the ceramic core named pure ceramic or all-ceramic restoration. The ceramic core of the second type is composed of various ceramic materials-most notably doped alumina and stabilized zirconia, which are characterized by a high durability. All ceramic dental restorations have become widely popular because of their aesthetic properties, particularly their responsiveness to colouring and glazing, making them suitable for the aesthetic region of the mouth. Recently, emphasis has been placed on zirconia due to its superior mechanical properties [1,2].

At room temperature and up to 1170°C, Zirconia (ZrO2) exists in the monoclinic crystalline phase that transfer to tetragonal phase within the 1170–2370°C range, and subsequently to cubic up to the melting point. The transition from the tetragonal phase to the monoclinic phase upon cooling is accompanied by a significant volume increase (approximately 4.5%) at around 950°C. The tetragonal phase can be

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stabilized down to room temperature via the addition of stabilizing oxides such as CaO, MgO, Y2O3, and CeO2. When the tetragonal phase is retained and a crack forms in the zirconia because the mechanical stress, the crack's energy is absorbed by the tetragonal phase as it transforms into the monoclinic phase. This transformation results in the volume increase; consequently, the resulting monoclinic phase exerts a stress opposing the crack's path, thereby arresting its propagation. This mechanism of halting crack propagation is known as "transformation toughening." This phenomenon is responsible for superior mechanical properties of zirconia [3]. Yttria (Y2O3) is typically preferred for preparing Yttria-Stabilized Zirconia (YSZ) because its ability to stabilize the material entirely—rather than only partially—in the tetragonal phase, thereby yielding the highest mechanical properties for the zirconia [4].

Research has demonstrated that zirconia exhibits a longer lifespan before failure compared to other dental ceramics, such as alumina [5–10]. Furthermore, zirconia has bio compatible properties with jawbone—enhancing osseointegration—and its ability to reduce bacterial adhesion on its surface compared to other materials like titanium [11–12].

On the other hand, the introducing of intraoral scanners facilitates obtaining a digital model of the mouth, and consequently the high-precise fabrication of bridges and crowns. This advancement was realized through the implementation of integrated CAD/CAM systems [13]. Indeed, the availability of these systems has encouraged dentists to involve all-ceramic restorations and prompted manufacturers to produce crystalline ceramics for this purpose—offering an alternative to glass-based ceramics, which possess inferior mechanical properties [14]. To address the problem of limited translucency in zirconia ceramics, small amounts of trivalent oxides (such as Al2O3, Sc2O3, Nd2O3, and La2O3) have been added to create a transparent phase at the grain boundaries, yielding a zirconia ceramic with a translucent appearance [15].

Although superior mechanical properties have been established by CAD/CAM-fabrication of zirconia bridges and crowns, the structural integrity of thin margins under mechanical loading remains a key concern for dentists. Naturally, zirconia margins demonstrate a higher capacity to withstand mechanical loads compared to other kinds of ceramic [16]. Practical data have also shown that zirconia crowns can endure the high compressive forces associated with mastication [17]. Nevertheless, operators continue to report cases of brittle fracture in this type of ceramic [18]. In clinics, the ceramic thickness in the occlusal region for CAD/CAM-fabricated zirconia crowns is typically around 0.5 mm, while maintaining sufficient durability to withstand mechanical loads [19]. It has been reported that increasing the thickness from 0.5 mm to 1.5 mm triples the fracture resistance [20]. Furthermore, it has been repeatedly noted that CAD/CAM-fabricated zirconia crowns and bridges—particularly those stabilized with 3 mol% yttria—exhibit excellent resistance to chipping and fracture [21–23]. The designation 3Y-TZP is used to denote 3 mol% yttria-stabilized zirconia, standing for "3 Mole% Yttria-Stabilized Tetragonal Zirconia Polycrystal."

In this study, the compressive strength—a key factor in enhancing durability—was significantly improved for 3Y-TZP zirconia. The material used was "GC Initial Zirconia Disks," produced by a leading medical materials company; these disks are formed using the Cold Isostatic Pressing (CIP) method [24]. This improvement was achieved by adding boron trioxide (B2O3)—an oxide used here for the first time to reinforce zirconia—combined with conventional pressing. The results exceeded expectations regarding compressive strength, while also increasing the percentage of elongation at maximum stress (strain %) and fracture toughness. This work falls within the research field of materials science and engineering; the results also encourage application in other engineering domains where mechanical properties are critical.

It is worth noting that boron oxide has been utilized in previous studies with materials other than the 3Y-TZP used here—examples include its addition to calcium-stabilized zirconia (CSZ) [25], mullite/unstabilized zirconia composites [26], porcelain [27], and porous unstabilized zirconia [28]. In the present study, however, boron oxide is added to 3Y-TZP for the first time.

Material and Methods

3Y-TZP material produced by GC manufacture was supplied in form of compacts (or blanks). These compacts were milled to obtain stabilized zirconia powder, facilitating the subsequent addition of other materials. A planetary ball mill was used for the milling process to produce a fine powder. A powder with an average particle size of 1236.8 nm (1.2368 µm) was obtained, as shown in Figure 1; this represents the smallest particle size achievable with the available milling method, given the high mechanical properties of stabilized zirconia. This milled powder was adopted as the initial material to ensure consistent pressing conditions for both the standard sample and the specimens containing additives.

The used additive material was milled boron oxide (B2O3) that passed through a 53 µm sieve. It is obtained by melting boric acid (H3BO3; 99.5% purity; BDH, USA) at 450°C, with a weight percentages of addition were 1 wt.% and 2 wt.%.

Preliminary experiments were conducted to define the optimal ratios of boron oxide addition by evaluating the achieved densities after sintering, which serve as an initial indicator of the microstructural integrity of the sintered samples. Accordingly, the samples were prepared as shown in Table 1.

To obtain a preferred agglomerated powder for press-forming operations, an alcoholic solution containing 2 wt.% of polyethylene glycol (PEG) was added to work as a binder and agglomeration aid. Specimens were formed by pressing in a tool steel mold at 400 kPa. The inner surface of the mold was coated with a thin layer of low-density paraffin wax to prevent friction between the compacted material and the die wall before filling the mold with powder to obtain uniform compaction. A hydrofix press (Bego, Germany) was used for the compaction process.

A specific sintering program to the manufacturer of the used stabilized zirconia [24] was applied, as detailed in Table 2.

A detailed flowchart of the manufacturing process is presented in Figure 2.

For particles size analysis, a laser particle size analyser (Brookhaven NanoBrook 90 Plus, USA) was used; the findings are presented in Figure 1. Density was measured using a four-decimal-place balance (Furi, China) via the Archimedes method; notably, the specimens exhibited identical weights before and after water immersion, demonstrating the absence of open surface pores. For sintering, a furnace (Ceramill Therm, Austria) was used, programmed according to the manufacturer's "GC" cycle as detailed in Table 2.

Compressive strength was measured using a universal testing machine (Laryee, China). The samples were cut into cubes with an initial side length of 1 cm, resulting in final cubic specimens with a side length of approximately 0.95 cm. Fracture toughness was evaluated using an Izod impact tester, and the microstructure of the fracture surface was examined via a scanning electron microscope (Angstrom Advanced AIS2300C SEM, USA). Additionally, an X-ray diffractometer (Shimadzu XRD 6000, Japan) was used to identify crystalline phases.

Appendix 1 illustrates the procedural steps, documented with photographs.

Results and Discussion

Density Measurements

Figure 3 illustrates the measured density values of the sintered samples, expressed as a percentage of the theoretical density of zirconia (5.68 g/cm³). The reference sample represents a 0% addition of boron oxide. An

Vol 14, No 3 (2026) DOI 10.5195/d3000/2026.1654

increase in density is observed with the addition of boron oxide, reaching a high value of 97.71% of the theoretical density at a 2% of boron oxide addition. However, adding more than 2% results in a decline in both density and compressive strength; this is attributed to the presence of an excessive amount of the glassy phase, which adversely influences the microstructure.

Identification of Crystalline Phases

To identify the crystalline phases resulting from the addition of boron oxide, X-ray diffraction (XRD) measurements were conducted on the standard sample and the specimen containing 2% boron oxide, as observed in Figure 4. The German-made" software "Match is widely regarded as the best toolfor the analysis of crystalline phase was used todetermine any new phases formed due to the addition of boron oxide. Both samples exhibited the crystalline phase [(ZrO2)0.95(Y2O3)0.05] corresponding to the 3Y-TZP material used, in accordance with the ,COD (Crystallography Open Database Lithuania) system utilized by the software. The specific entry number for this stabilized zirconia in this system is [96-152-1475], system as indicated in the figure. Notably, no corresponding phase was found in the available JCPDF (Joint Committee for Powder Diffraction Files, USA) database; instead, only the ZTP phase (without yttria addition) was found under entry (JCPDF 00-050-1089). This discrepancydevelops due to the COD database is updated much more frequently than the JCPDF database and is available free of charge upon release.

The figure reveals that the only difference is an increase inthe intensity of background particularly in the low-angle region indicating the presence of a glassy phase. This phase corresponds to boron oxide which is typically amorphous. In other words, the addition of boron oxide did not produce a new crystalline phase; rather, XRD analysis revealed the presence of a glassy phase. The glassy phase is expected tohelp in two ways: filling the voids within the microstructure and facilitating the rearrangement of grains, thereby helping to achieve a densely packed microstructure; this explains the increase in density upon the addition of boron oxide, in accordance with the mechanism described in reference [29].

Compressive Strength and Fracture Toughness

To determine the compressive strength, percentage elongation at maximum stress, and fracture toughness—key specifications for the practical application of stabilized zirconia in dental bridges and crowns—compressive strength and fracture toughness tests were conducted. The resulting values are presented in Table 3.

The table above shows that the addition of 1% boron oxide increased compressive strength by approximately five folds. Adding 2% boron oxide increased compressive strength more than ten folds, reaching the testing machine's design limit (100 kN) without causing specimen failure. Fracture toughness also increased approximately five folds (measured in kgf·m, representing the energy required to fracture a unit area). This remarkable surge in properties—which will be discussed further below—significantly reduces the risk of bridges and crowns shattering during chewing or accidental drops. Additionally, the percentage of elongation at maximum stress increased progressively with boron oxide additions. This increase is highly significant, as it allows for greater flexibility when bending the margins of bridges and crowns during removal or re-fitting, preventing the delicate edges from shattering. Plots of the actual laboratory-conducted compression tests are provided in Appendix 2.

Figure 5 shows sintered specimens of the fabricated compacts, which were used to produce a dental model via CAD-CAM, as detailed below.

Figure 6 presents a photograph of the specimen containing 2% boron oxide, both before and after the compression test. The image highlights two key observations: First: The zirconia surface retained its luster despite the 2% boron oxide addition; the gloss—derived from the small amount of alumina in the composition—remained unaffected. Secondly: No visible cracks were observed on the surface of the specimen after it underwent the compressive strength test. In other words, the specimen retained its aesthetic properties following the compressive strength test.

Microstructure

To get better understanding to the enhanced compressive strength and fracture ,toughness the microstructure was examined using a scanning electron microscope (SEM). Figure 7 shows themicrostructure of fracture surface of the standardsample and that of the sample with1% boron oxide added. A quick comparisonreveals a clear presence of pores ,in the microstructure of the standard sample whereas these pores are absent in the sample containing 1% boron oxide. This aligns with the density and X-ray diffraction (XRD) analysis results, indicating that the addition of boron oxide filled pores and induced grain rearrangement within the microstructure. The findings are consistent with the conclusions presented in [29].

The increase in the percentage of elongation at maximum stress from 2.990 % to 3.396% (approximately 14% increase), upon adding 2% boronoxide is considered a natural consequenceof the improved resistance tothe applied stress, resulting from the achievement of a dense pore-free microstructure.

It was not possible to obtain an image of the fracture surface for the sample containing 2% boron oxide, as it did not fail under the maximum force capacity of the testing machine; however, the sample's external surface was examined using a scanning electron microscope, and images at two different magnifications are presented in Figure 8. The images reveal a surface free of visible defects, although some roughness is evident—an unavoidable feature when examining the surface at magnifications exceeding 50× or 100×. Nevertheless, this roughness does not detract from the aesthetic quality, given the luster visible to the naked eye, as shown in Figure 6.

Figure 9expresses an image of a tooth fabricated from a compacted block (measuring cm × 1 cm × 6 cm) that underwent CAD/CAM 1 shaping and subsequent sintering, as described in the experimental section. The tooth is designated as number 26—the upper left first molar (where the digit '6' indicates the tooth position and '2' indicates the quadrant) according to the FDI World Dental Federation notation system.

The data of this work demonstrates that the selected boron oxide additive effectively increased post-sintering density and enhanced the integrity of the microstructure without inducing the formation of new crystalline phases. Instead, the boron oxide acted as a glassy phase that filled pores and facilitated ;grain arrangement within the microstructure this, in turn led to a significant increase in compressive strength without compromising the surface luster of the sintered specimen.

It is evident from the above experiment that the compressive strength was improved without the use of nanomaterials. While the standard practice involves using particles smaller than 100 nm, the starting material here was a powder with a particle size ranging from 700 to 2,000 nm (Figure 1)—averaging 1,236.8 nm (1.2368 µm). Notably, this good performance was achieved without incorporating highly advanced, expensive additives—such as carbon nanotubes or graphene—commonly used for this purpose. Furthermore, the preparation of the compacts did not rely on costly techniques like Cold Isostatic Pressing (CIP)—a method employed by GC [24], but rather on a standard pressing at a moderate pressure of 400 kPa. Also, the manufacturer's (GC) sintering protocol was not altered; this facilitates a

smooth transition to the new material while achieving high compressive strength and improved percentage elongation at maximum stress. Consequently, this minimize losses caused by edge chipping or fracturing of bridges and crowns—whether resulting from long-term mastication, accidental dropping, or handling during dental placement.

Applications

3Y-TZP (Yttria-stabilized tetragonal zirconia polycrystal) is highly regarded for applications requiring superior mechanical properties. In dentistry, zirconia compacts are used—shaped via CAD/CAM systems and subsequently sintered according to the manufacturer's specific protocols—to achieve the final product's required mechanical specifications and dimensions. However, the complex geometries required for dental bridges and crowns with thin margins can compromise mechanical performance; dentists are often constrained by specific design limitations dictated by each patient's unique clinical ,situation. This can lead to operational issues such as the cracking or fracturing of thin margins—particularly under forces exerted during mastication, cementation, or the application of significant pressure by the dentist during intraoral placement. This study successfully produced zirconia compacts with ,enhanced mechanical properties characterized bya significantly higher compressive strength and improved .percentage elongation at peak stress Consequently, this reduces the risk of material loss due to fractures of margins in zirconia bridges and crowns.

Furthermore , this work facilitates the production of dental restorations with thinner ,margins—a significant advantage for dentists as it minimizes potential gingival damage and reduces the need for aggressive tooth reduction during preparation. Creating thicker ,margins compromises the crown's aesthetics ,leads to food accumulation around the edges and causes inflammation and bleeding in the adjacent gums during brushing—ultimately resulting in gum recession and an unsightly appearance. Moreover, increasing margin thickness necessitates removing more tooth structure, which negatively impacts the tooth's health.

Additionally, improving mechanical properties did not rely on obtaining nanopowders of stabilized zirconia or incorporating expensive additives like carbon nanotubes, or graphene—as seen in recent research. Instead the approach utilized a single, suitable, and low-cost additive: boron oxide. This reduces the production cost of stabilized zirconia and encourages the development of a local industry for this product.

Advantages

1- A significant increase in compressive strength and a notable improvement in the percentage of elongation at maximum stress were achieved. This reduces the risk of material loss caused by the fracturing of thin margins in stabilized zirconia bridges and crowns.

2- Tooth integrity is preserved by minimizing the amount of tooth structure removed during bridge or crown preparation. The improved properties mentioned in point 1 allow for the fabrication of thinner margins—meaning less tooth reduction—whereas thicker margins required to maintain mechanical strength) without such additives) would necessitate removing more tooth structure, thereby compromising dentalhealth.

3- The aforementioned specifications were ,achieved by adding low-cost boron oxide .avoiding the need for expensive additives Furthermore, there is no need to use expensive nanopowders of stabilized zirconia, nor is there a requirement for costly compaction ;techniques such as cold isostatic pressing instead, conventional pressing is employed Additionally, the standard sintering program for stabilized zirconia remains unchanged.

Claims

Claim 1: Preparation of stabilized zirconia compacts suitable for dental bridges and crowns with thin margins, exhibiting superior mechanical properties. This is achieved through the addition of boron oxide; optimal results—specifically a very significant increase in compressive strength and a notable increase in the percentage of elongation at maximum stress—were obtained with a 2% addition. Consequently, there is no need for expensive additives to enhance mechanical properties, nor is there a requirement for stabilized zirconia nanopowders, cold isostatic pressing, or modification of the standard sintering program for stabilized zirconia.

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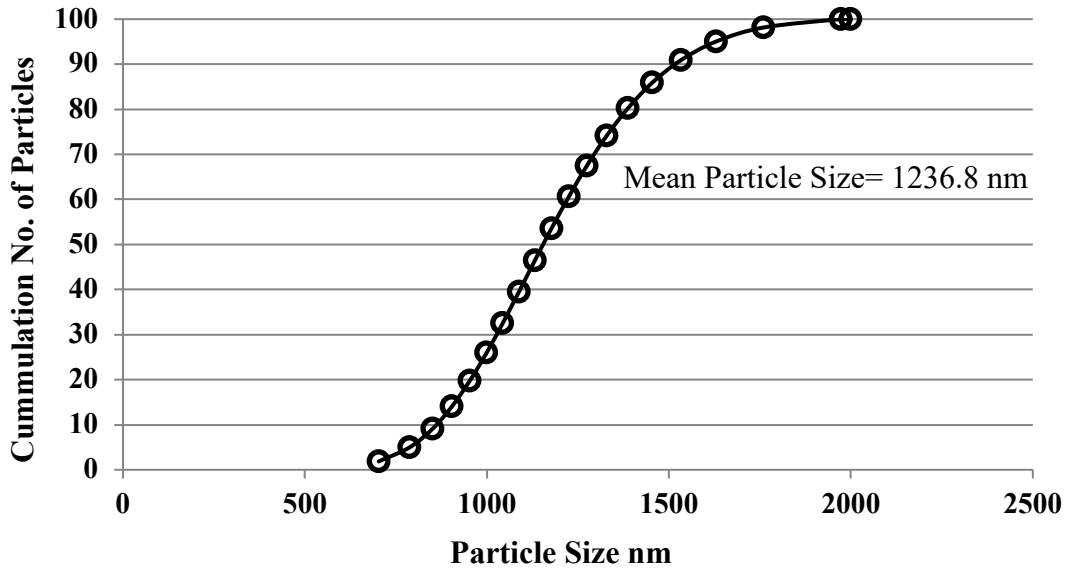


Figure 1. Cumulative particle size of the milled zirconia.

Table 1. Boron oxide addition ratios to stabilized zirconia.

Boron Oxide	Stabilized Zirconia	Sample code
-----	100 wt.%	Control
1 wt.%	99 wt.%	ZRB1
2 wt.%	98 wt.%	ZRB2

Table 2. Sintering program used according to GC company documentation [24]—the source for the standard sample—which matches the program set for the company's own sintering furnace.

	Heat Ramp1	Heat Ramp2	Hold	Cool Ramp3
Temperature °C	1000	1450	1450	1000
Time (hour)	2	4.5	2	1

Milling the GC company disc using a planetary mill.
Analysing the particle size of the resulting powder using a laser diffraction instrument.
Weighing the resulting powder and dividing it into six equal-weight portions.
Adding 0%, 1%, and 2% B2O3 to the 3Y-TZP powder.

↓

Mixing polyethylene glycol (PEG) with alcohol at a 2:1 ratio to prepare a binder for mixing with the powders prior to pressing.

↓

Coating the inner surfaces of the sample-pressing mold with light paraffin oil to serve as a mold lubricant.
Note: The mold dimensions are 1 cm × 1 cm × 6 cm, consistent with the specifications of most manufacturers of these compacts.
Pressing the samples using a uniaxial pressing method at a pressure of 400 kPa.

↓

Sintering the samples with a ramped heating profile up to 1450°C over a 9.5-hour cycle, as detailed in Table 2 (following .(the standard GC company sintering program

Figure 2. Detailed flowchart of the compact manufacturing process.

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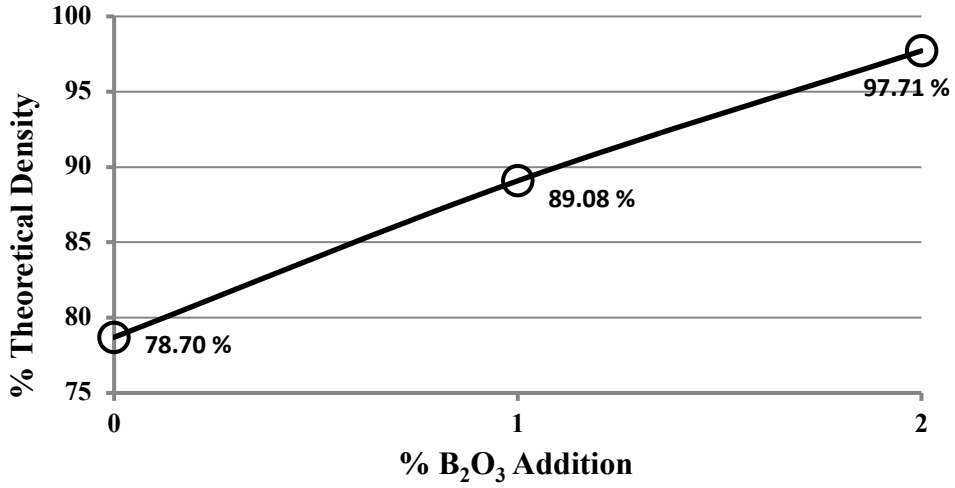


Figure 3. Variation of density with additives for sintered samples.

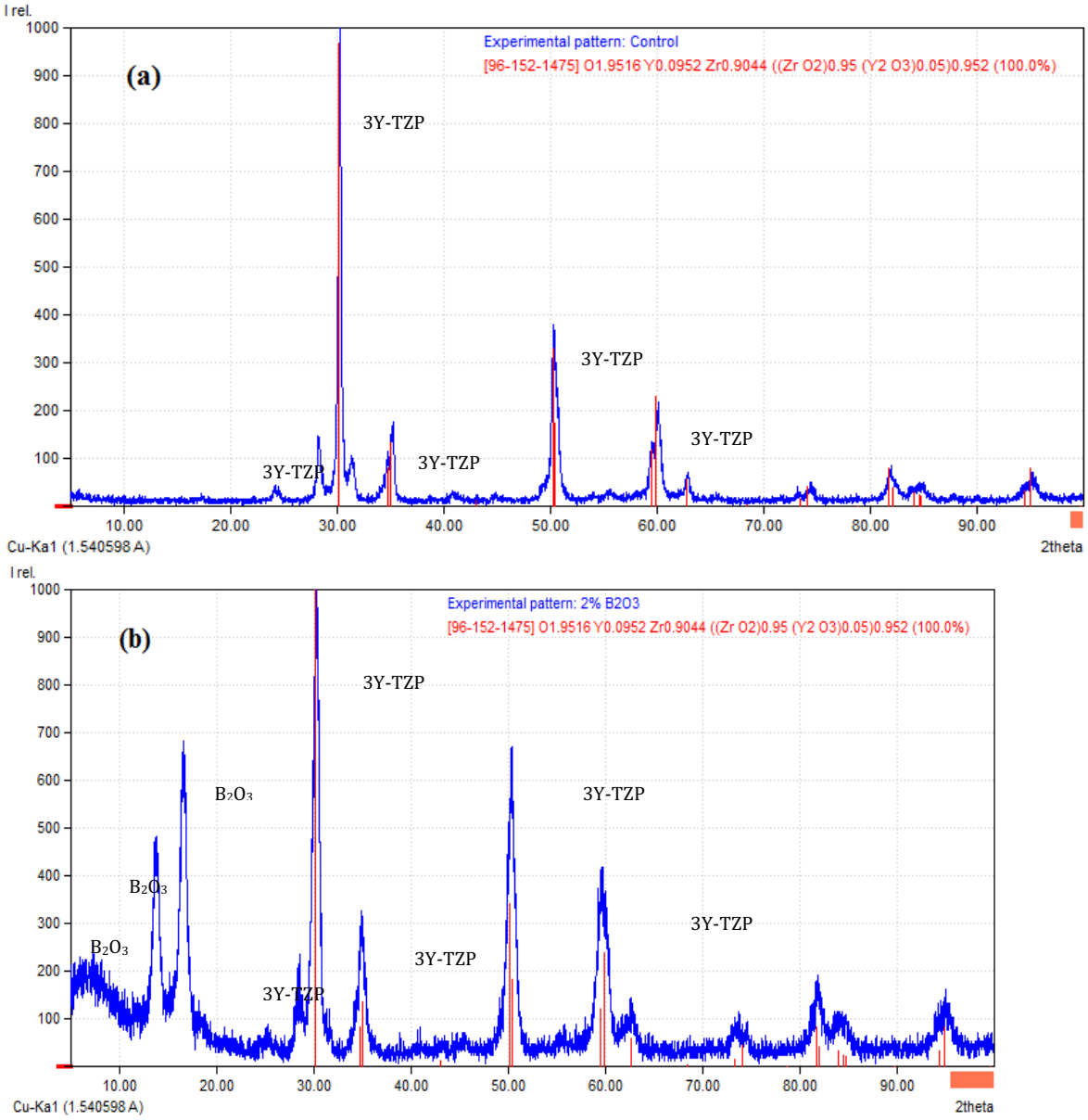


Figure 4. X-ray diffraction patterns for (a) the standard sample and (b) the sample with the addition of 2% B2O3. The increased background—attributed to the glassy phase—and the peaks corresponding to B2O3 indicate its partial crystallization [30].

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Table 3. Compressive strength, applied load, elongation percentage at maximum stress and fracture toughness.

Elongation percentage at maximum Stress %	Maximum Stress kN	Compressive strength MPa	Fracture toughness Izod Kgf.m	Code
2.990	14.56	100.873	10.59	Control
3.008	48.42	527.106	24.24	ZRB1
3.396	98.4 *	1180.847	49.57	ZRB2
*At this point, the experiment was stopped from the employer to maintain the device and the sample did not fail. No machine was found to provide force more than 100kN.				

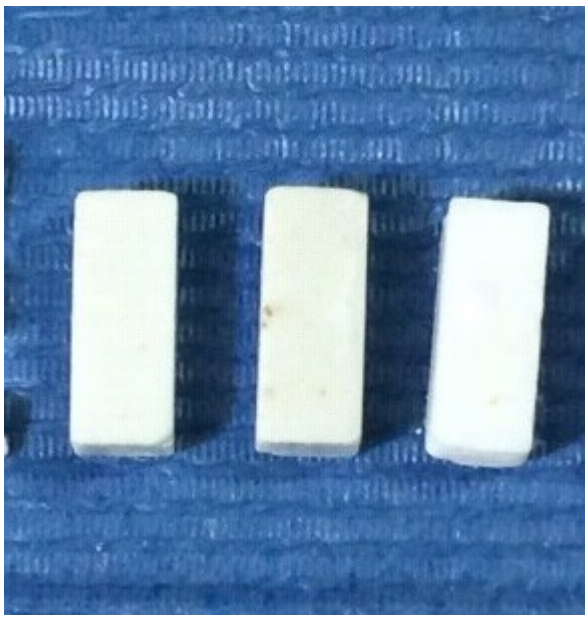


Figure 5. The compressed sample of zirconia fixed with the addition of 2% boron oxide and sintered according to Table 2.
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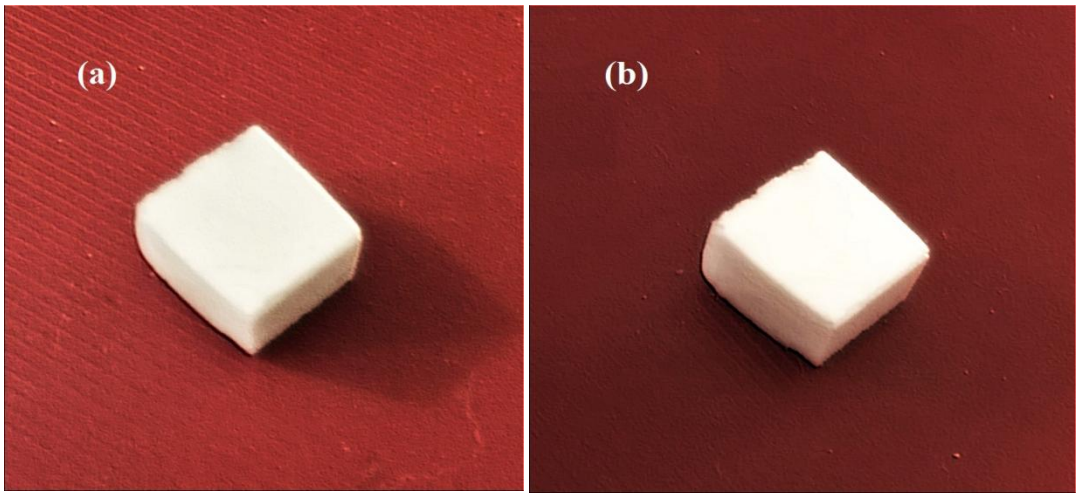


Figure 6. Sintered, cube-shaped sample of stabilized zirconia with the addition of 2% boron oxide: (a) before the compressive strength test and (b) after the compressive strength test.

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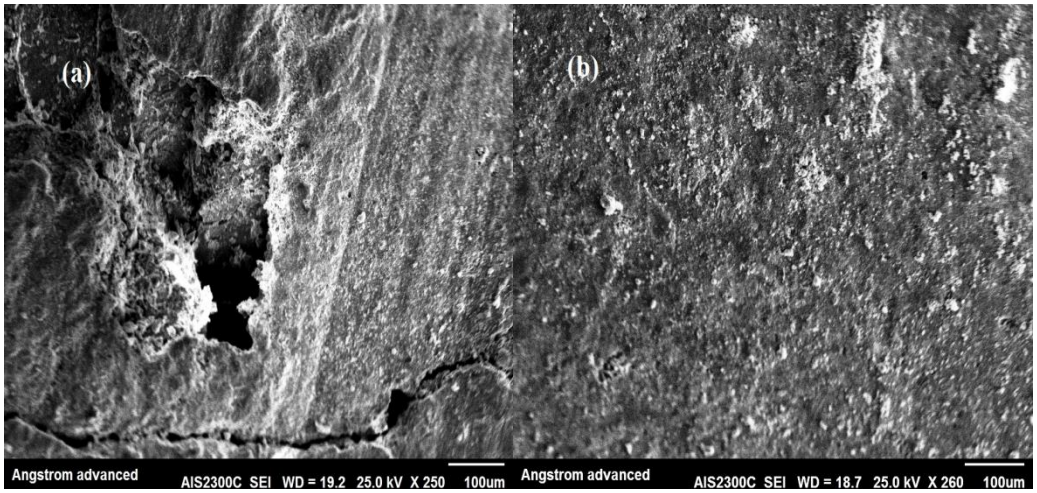
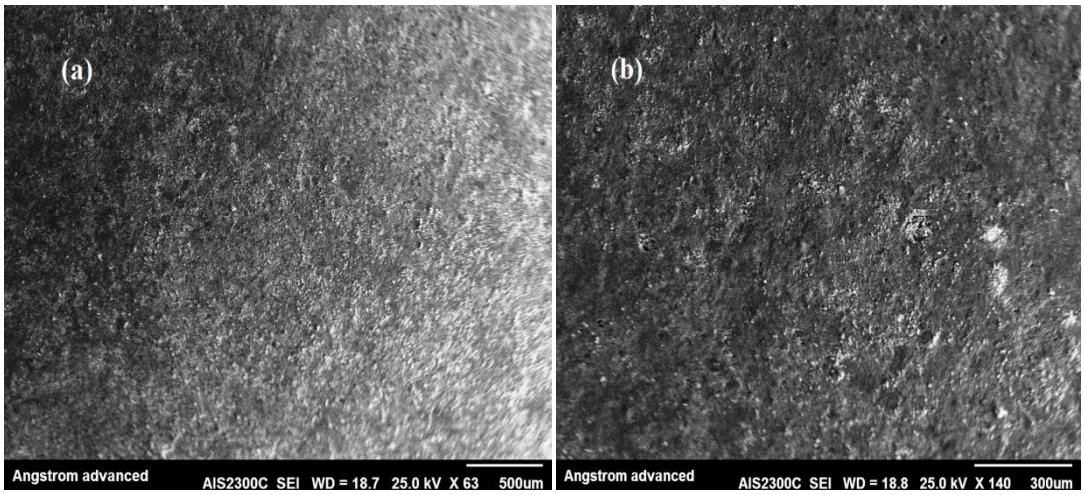
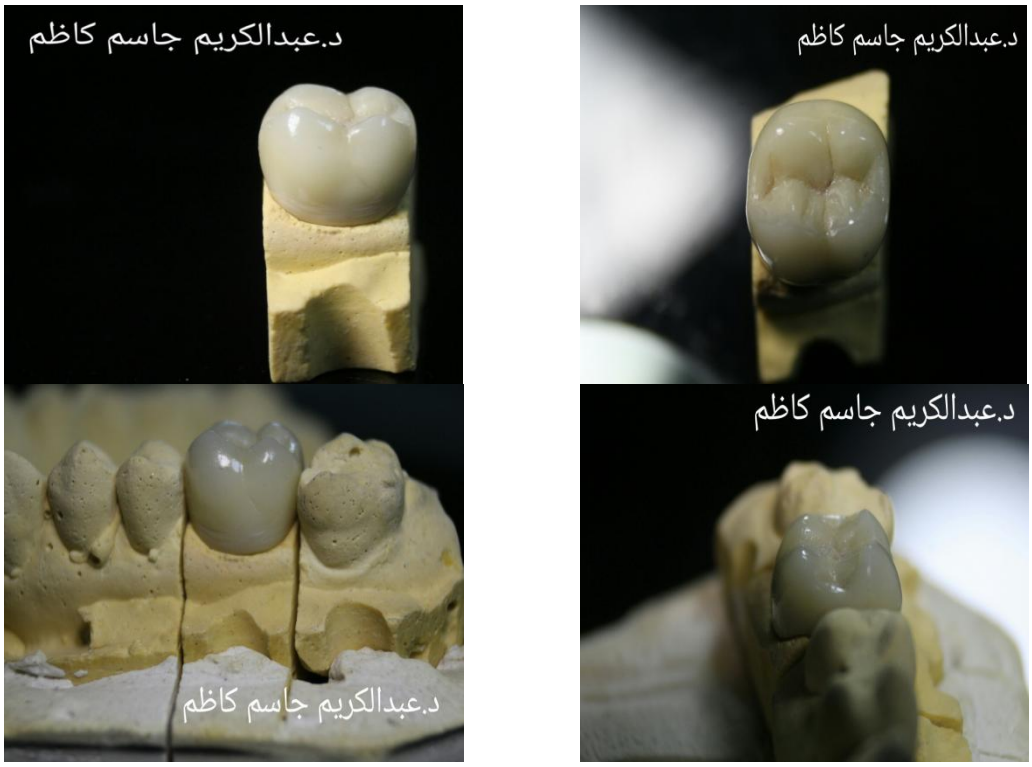


Figure 7. Scanning electron micrograph of the fracture surface for (a) the standard sample and (b) the sample with the addition of 1% boron oxide.



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Figure 8. Scanning Electron Microscope (SEM) images of the sample surface with the addition of 2% boron oxide: (a) 500 µm scale bar and (b) 300 µm scale bar.



Appendix No. 1
Practical steps documented with photographs



Zirconia blank from GC

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Grinding device

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Zirconia powder after mold milling





Laser diffraction particle size analyzer with a zirconia powder sample

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Loading a zirconia powder sample into the laser diffraction

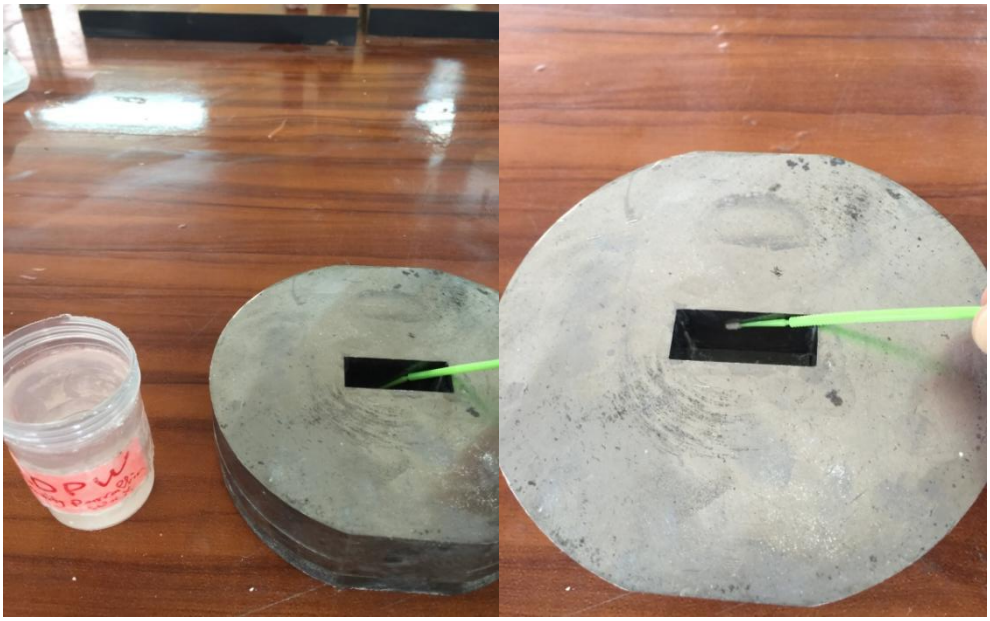


Materials used in the practical side, from the left; Ground zirconia powder, polyethylene glycol powder, light paraffin, boric acid

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.Weighing the required amounts of zirconia powder



.Coating the inner surfaces of the compression mold with light paraffin

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Filling the pressing mold with zirconia powder

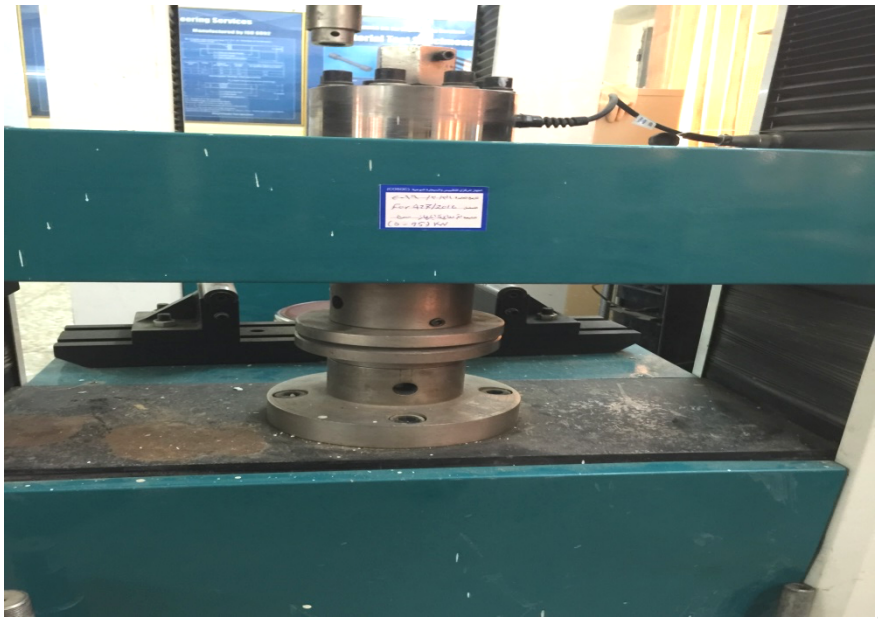


.Pressing device

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Sintering furnace. It is from GC, the same manufacturer as the zirconia discs, and the sintering process was carried out using



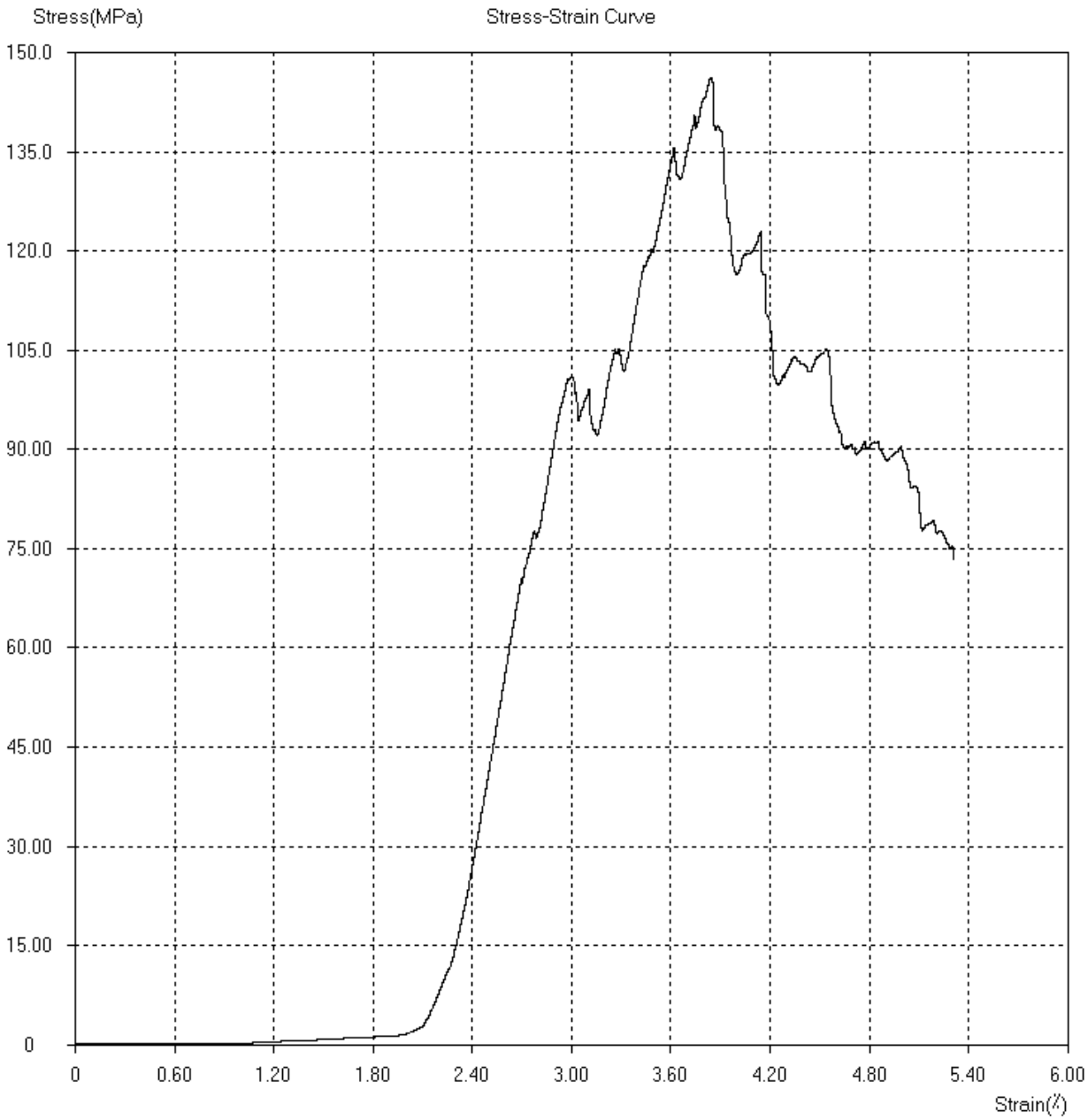
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.Compressive strength testing apparatus

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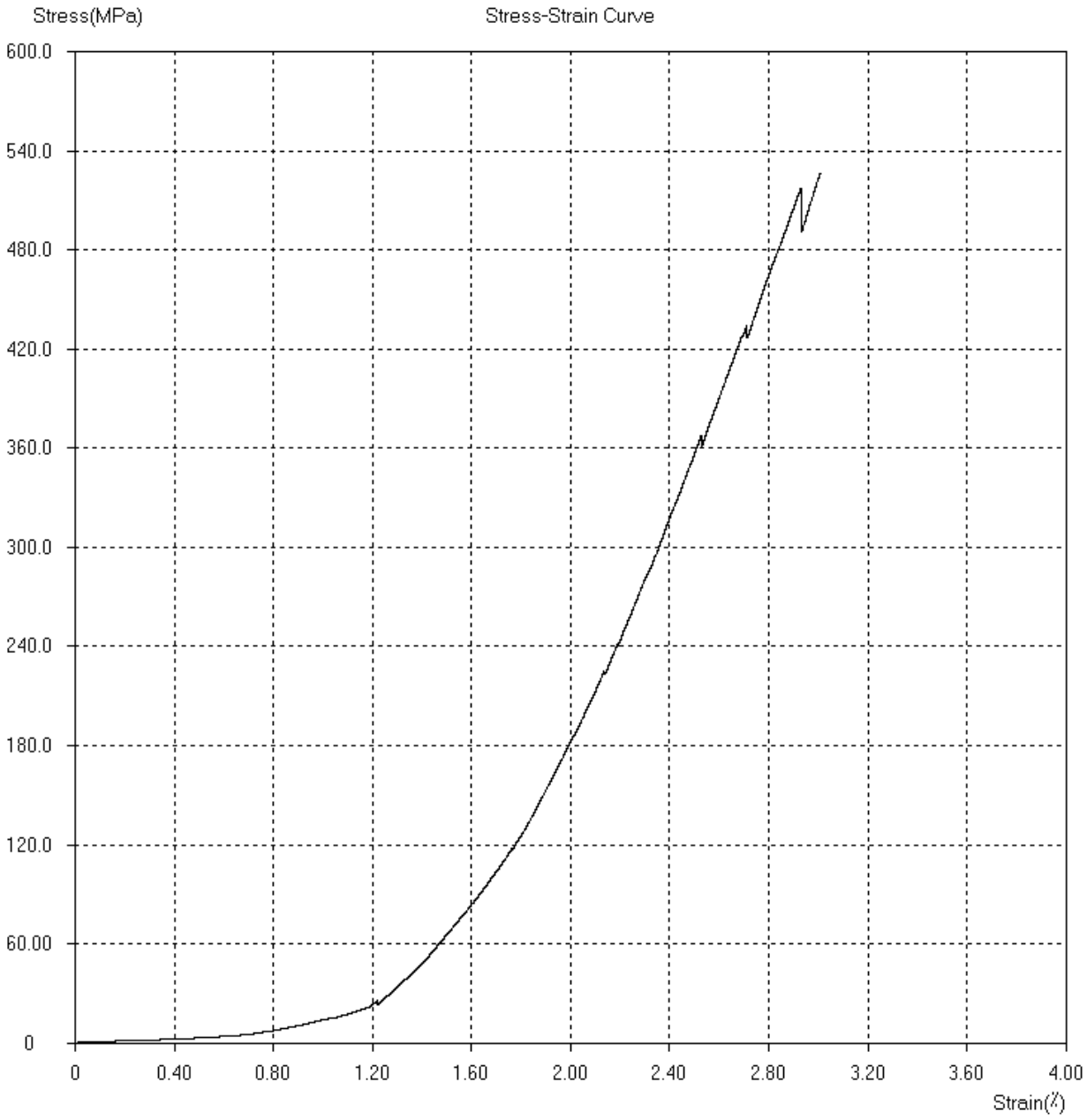
Appendix No. 2

Actual compression plots obtained in the laboratory



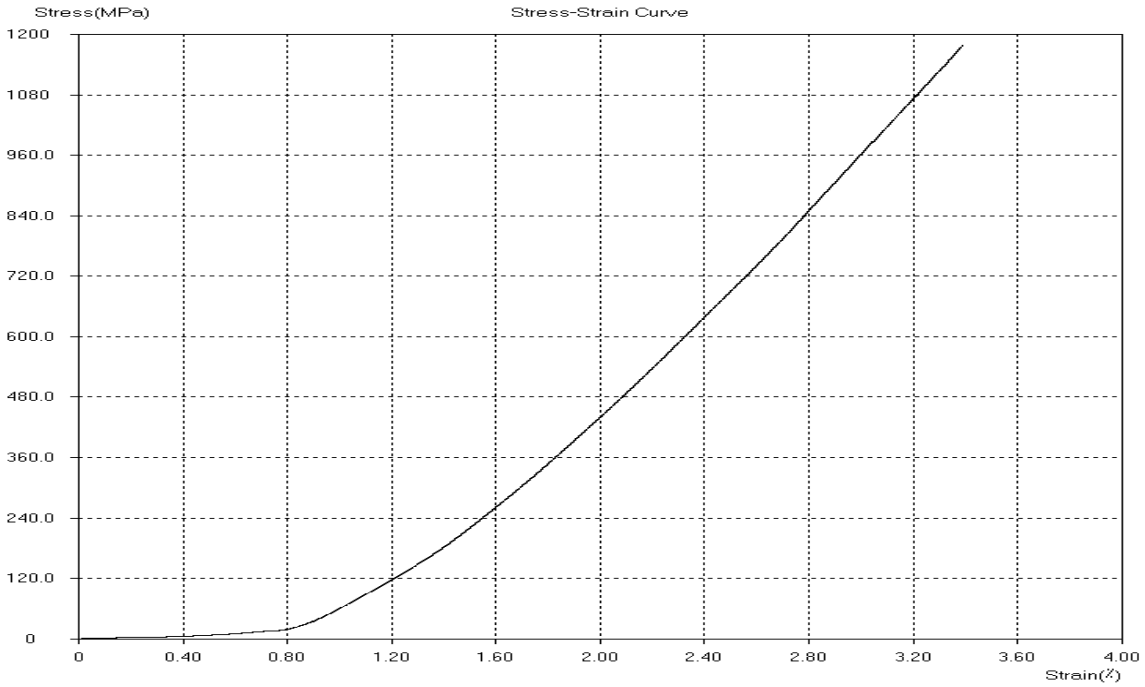
Actual compression plot obtained in the laboratory for the standard sample

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Actual compression plot for the stabilized zirconia sample
.with the addition of 1% boron oxide

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Actual compression plot for the stabilized zirconia sample
.with the addition of 2% boron oxide