

Article

Comparative Study of the Influence of Heat Treatment on Fracture Resistance of Different Ceramic Materials Used for CAD/CAM Systems

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Abstract: The aim of this study was to compare the influence of heat treatment on fracture resistance (FR) of different ceramic materials used for CAD/CAM systems. **Methods:** Eighty monolithic restorations were designed using the same parameters and milled with a CAD/CAM system (CEREC SW 5.0, PrimeMill, Dentsply-Sirona™, Bensheim, Germany), forming five study groups: Group 1 (n = 10), CEREC Tessera (Dentsply-Sirona™, Bensheim, Germany) crystallized (CCT), Group 2 (n = 10), CEREC Tessera uncrystallized (UCT), Group 3 (n = 20), Emax-CAD (Ivoclar Vivadent, Schaan, Liechtenstein) (CEC), Group 4 (n = 20), Vita Suprinity (Vita Zahnfabrik, Bad Säckingen, Germany) (CVS), and Group 5 (n = 20) Cameo (Aidite, Qinhuangdao, China) (CC). **Results:** The average FR was similar for CCT, CC, and CEC at above 400 N, while CVS and UCT had the lowest values at 389,677 N and 343,212 N, respectively. **Conclusion:** Among the three ceramic materials that exhibited an FR above 400 N, CCT was considered the first recommended choice for CAD/CAM systems. This material not only demonstrated the highest FR but also exhibited outstanding consistency in the related measurements without the presence of outliers. Although the CC material showed high FR, its high dispersion revealed inconsistencies in the repetitions, suggesting caution in its use.

Keywords: ceramics; crystallization; mechanical characterization; CAD/CAM materials; properties; thermal influence



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1. Introduction

The emphasis on esthetics in dental restorations has concomitantly driven the rapid evolution of metal-free ceramic materials [1,2], used and valued for their outstanding mechanical and optical properties regarding dental restorations [3–6]. As an example, lithium silicate-based (SL) glass ceramics designed for dental CAD/CAM systems have been introduced in the market [7]. These SL-based ceramics are mainly composed of Li₂O and SiO₂. Depending on the predominant phase during their crystallization, they are classified as “lithium disilicate” (Li₂SiO₂₅), “lithium silicate” (Li₂SiO₃), or “(di)lithium silicate” (the latter regarding those with significant proportions of both former phases) [8–10]. In this study, after an exhaustive review of the ceramic materials for CAD/CAM systems currently available in the dental field, they were classified into four types as detailed in Table 1 and associated into five study groups; see Table 2.

Table 1. Sample and manufacturer details of the four types of CAD/CAM ceramics.

Name of Material	Manufacturer	Lot Number	Composition
Cerec Tessera	Dentsply Sirona™/Germany	16015140	Li ₂ Si ₂ O ₅ : 90% Li ₃ PO ₄ : 5% Li _{0.5} Al _{0.5} Si _{2.5} O ₆ (virgillite): 5%
Emax CAD	Ivoclar Vivadent™/Liechtenstein	6788824	SiO ₂ : 57–80% Li ₂ O: 11–19% K ₂ O: 0–13% P ₂ O ₅ : 0–11% ZrO ₂ : 0–8% ZnO: 0–8% Coloring oxides: 0–8%
Vita Suprinity	Vita Zahnfabrik/Germany	7971329	SiO ₂ : 56–64% Li ₂ O: 15–21% ZrO ₂ : 8–12% P ₂ O ₅ : 3–8% K ₂ O: 1–4% Al ₂ O ₃ : 1–4% CeO ₂ : 0–4% Pigments: 0–4%
Cameo	Aidite/Singapore	9180451	50–70% SiO ₂ ; 19–20% Li ₂ O; 0–13% K ₂ O; 0–11% P ₂ O ₅ ; 0–5% ZrO ₂ , 0–8% ZnO; 0–11% others + coloring

Table 2. Study groups, ceramics and samples.

Groups	Ceramics	Samples
Group 1	Cerec Tessera (CCT)	10
Group 2	Cerec Tessera (UCT)	10
Group 3	Emax Cad (CEC)	20
Group 4	Suprinity (CVS)	20
Group 5	Cameo (CC)	20

UC: Uncrystallized; C: Crystallized.

There is an inverse relationship between mechanical and optical properties. Materials with higher crystalline content are characterized by higher mechanical properties, but higher opacity, while higher vitreous content results in higher translucency, but is characterized by lower mechanical performance. It should be considered that translucency is critical in material selection and is of great clinical importance, also it is advantageous for materials to have ideal mechanical performance, so the field is constantly evolving [11,12].

When LDS ceramics are produced for restorative use, additional steps are required for their milling, such as crystallization, because there are small crystals that, when exposed to heat, enlarge and form colonies that intertwine among themselves. This causes material microstructure similar to a mesh, resulting in a dendritic morphology in the form of a tree or sheaf with important ramifications. These acicular structures help to achieve high resistance and tenacity to fracture in glass–ceramic [13–15].

One of the most recent iterations of glass matrix ceramics is called CEREC Tessera™ (Dentsply Sirona, Germany). It is characterized as an advanced SL with a glass content of 40–45% and a submicron particle size of ~0.5 µm; it is composed of ~40% lithium disilicate crystals, 5% lithium phosphate and 5% virgillite crystals, which are small (<100 nm) lithium aluminum silicate crystals present in the form of platelets. When using this material, the manufacturer recommends subsequent milling and surface glazing, in addition to subjecting the restoration to heat treatment AHT of 4 min and 30 s to optimize the crystalline structure by forming new virgillite crystals; this nucleation guarantees an increase in its mechanical properties, 700 MPa FR, and better physical properties by maximizing the presence of crystals and the generation of compressive stress around them [16–18].

The inclusion of virgillite crystals, proposed to improve translucency [19], suggests the manufacturer to consider a UCT variant with the aim of improving machinability during milling. It is crucial to point out the importance of high precision in this process, as low precision could lead to errors in dental prostheses, generating marginal discrepancies between the crown and the tooth, potentially leading to clinical failure [14,20].

The FR of a metal-free ceramic material is determined by the analysis of its mechanical properties. To elaborate, the masticatory loads in the oral cavity of humans act with various

dimensions and directions [21–23]. Thus, ceramic materials required to withstand these loads experience forces including compression, tension, and shear. Such varying stresses result in more complex loading patterns and, hence, it is possible for a restorative material to fail even under lower-than-expected loads. Therefore, FR stands out as a factor integral to the determination of the longevity of restorations [24–27]. Indeed, it is essential to understand that FR is intrinsically linked to the composition and microstructure of a given dental restorative material [28,29].

It is therefore important to examine the abovementioned mechanical properties of a restorative material under thermal influence in a furnace recommended by the manufacturer and under the given AHT program. Notably, incorrect temperature increases or mismanagement of the cooling rate of a material could influence both the mechanical and optical characteristics of an AHT program [30,31]. Moreover, the internal fit and marginal accuracy could be affected by the crystallization of crystalline-reinforced ceramic materials [32–35].

One of the objectives of the digital flow is to optimize clinical time and maximize the possibility of finishing a restoration in the course of a single appointment; thus, the time spent in the treatment of the restorative material is a variable that should be discussed and considered. Clearly, the less time taken to fabricate CAD/CAM ceramic restorations without affecting the mechanical properties, the more efficient the restorative process [36–40]. In this light, the aim of this study is to compare the influence of heat treatment on the fracture toughness of different ceramic materials used in CAD/CAM systems. The null hypothesis of this research states that there will be no significant differences in the fracture toughness of different ceramic materials used in CAD/CAM systems when subjected to different heat treatments.

2. Materials and Methods

2.1. Sample Processing

To prepare the samples, the model of an upper first molar designed for a full crown with a chamfer finish line was scanned for this study. The preparation was digitized using an intraoral structured light scanner (Primescan, Dentsply Sirona™, Bensheim, Germany). A full-volume restoration of 1.25 mm thickness was designed using the CAD software (CEREC SW 5.0, CEREC, Dentsply Sirona™, Bensheim, Germany); thereafter, the prepared blocks (Figure 1) were machined using a milling machine (PrimeMill, Dentsply Sirona™, Bensheim, Germany) and subjected to the manufacturer-recommended heat treatment (as detailed in Table 3) and (Figures 2 and 3), eventually forming five study groups (Table 2).

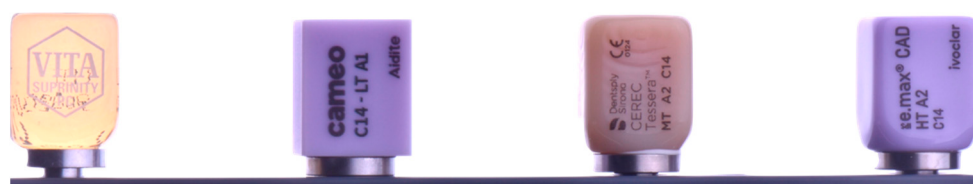


Figure 1. CAD/CAM ceramic blocks.

Table 3. CAD/CAM ceramics and manufacturer-recommended AHT.

Name of Material	Oven	Manufacturer	Heating Units	Time
Cerec Tessera	Programat P310	Ivoclar Vivadent™/ Liechtenstein	760 °C	2 min
Emax CAD			850 °C	7 min
Vita Suprinity			840 °C	8 min
Cameo			840 °C	6 min

The number of samples was based on various articles and assays used for this purpose [41–45].



Figure 2. CAD/CAM/SC ceramic blocks.



Figure 3. CAD/CAM/C ceramic blocks.

2.2. Thermocycling

All samples were subjected to a thermocycling process; in total, 5000 [6] cycles were used to estimate five years of oral conditions. Thermocycling was programmed with temperature extremes of 5 °C and 55 °C in distilled water (residence time: 25 s; pause time: 10 s) and performed on the computerized thermocycling unit (Thermocycler™, SD Mechatronik, Westerham, Germany).

2.3. Fracture Resistance Test

A cast-metal master die obtained from the study's initial scan of the original typodont was fabricated to support the try-in of each ceramic restoration specimen. Each specimen was subjected to a static load test at a speed of 0.5 mm/min in a direction parallel to the major axis of the tooth and with an initial preload of 10 Newtons (N) using a universal testing machine (Shimadzu, AGS X Series, Tokyo, Japan) equipped with a 20 kN load

cell. The load was applied using a hardened-steel pilot punch with a radius of 3 mm in the central pit of the restoration. All specimens were loaded to fracture and recorded in Newtons (N) by a software (Trapezium X Testing Software, Shimadzu, Tokyo, Japan) connected to the testing machine (Figure 4).



Figure 4. Loading of the specimen seated on the cobalt–chromium alloy abutment using a universal testing machine.

2.4. Fracture Mode Evaluation

After loading, the fracture surfaces of the specimens were observed and analyzed using a high-resolution stereomicroscope [Olympus; SZX7, New York, NY, USA].

2.5. Data Analysis

Data from each of the abovementioned groups were recorded in an Excel™ spreadsheet (Microsoft, Redmond, WA, USA). Subsequently, they were imported into a database in the SPSS 22 software (Statistical Package for Social Science, IBM Corporation, New York, NY, USA) for descriptive and inferential statistical analyses. For these purposes, nonparametric tests, such as the Kruskal–Wallis test, were used. To further explore the differences in data between the groups, multiple comparisons were performed using the Mann–Whitney U statistic.

3. Results

The descriptive statistics revealed that the maximum strain was reached by material CCT of $437,462 \pm 69.17$, followed by material CC $436,604 \pm 161.403$. They were followed by material CEC with a maximum strain of $434,968 \pm 88.019$ and moderate dispersion. In the fourth position came material CVS, which recorded a maximum strain of $389,677 \pm 73.85$. Finally, the material with the lowest maximum strain was UCT $343,212 \pm 25.143$ (Figure 5).

To further explore these differences that were observed, multiple comparisons were carried out using the Mann–Whitney U statistic, as evidenced in Table 4. This study found that there were statistically significant differences in terms of maximum compression between the UCT material and the CC, CCT and CEC materials (p -value < 0.05).

A fractographic analysis was carried out (Figure 6), where it was visualized that there was a small plastic deformation at the site of the load application, in which there was absorption of the deformation energy; this in Figure 6a,c. However, a zig zag brittle fracture typical of materials with crystalline structures occurred. The cracks followed the grain boundaries with irregular routes as seen in Figure 6b,d,e. These materials require greater strength for fracture due to their crystalline structure.

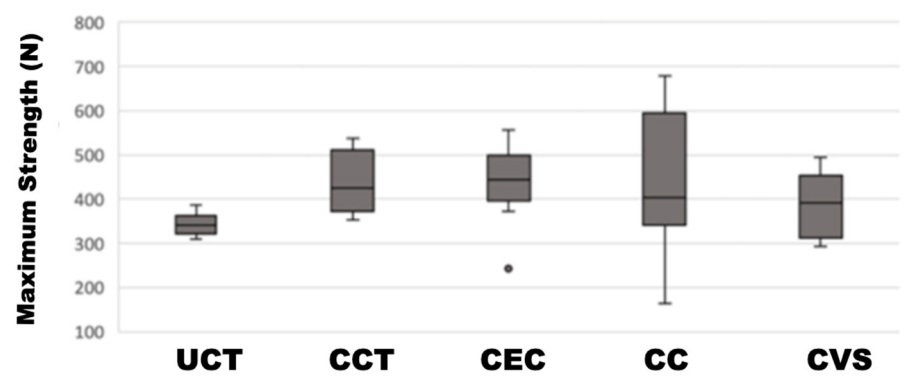


Figure 5. Box-and-whisker plot representing the data of the groups studied.

Table 4. Multiple comparisons: Mann–Whitney U test.

Sample 1–Sample 2	Test Statistic	Median	Sig.
Tessera uncrystallized–Cameo	−14,900	62.677	0.017 *
Tessera uncrystallized–Tessera crystallized	−17,189	83.924	0.008 *
Tessera uncrystallized–E-Max	−17,500	103.863	0.005 *

Note: Significance level of 5%. Shapiro–Wilk: p -value ≥ 0.05 ; Levene’s test: p -value < 0.05 ; Kruskal–Wallis H: p -value < 0.05 . * p -value < 0.05 .

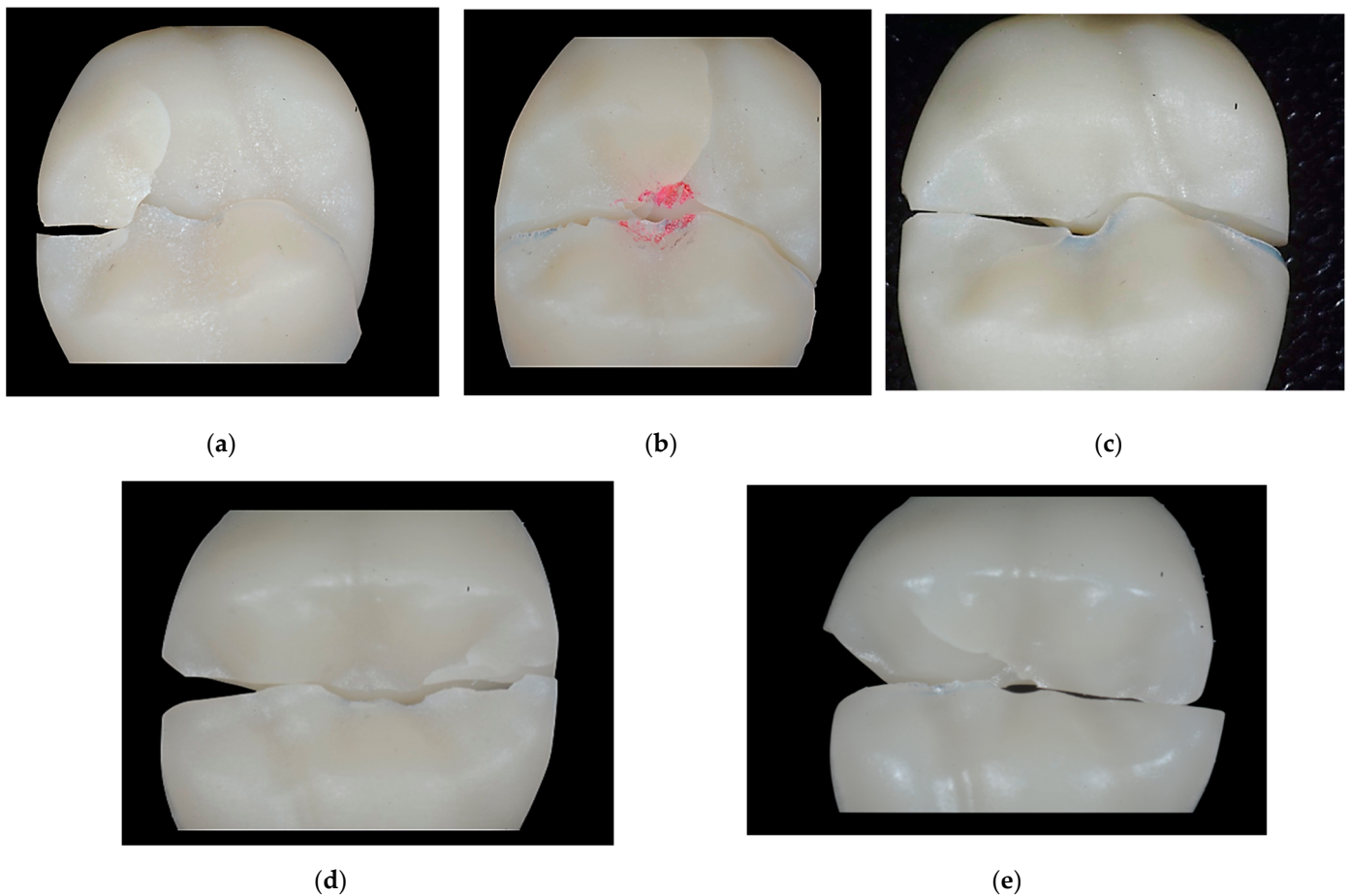


Figure 6. Images of the fracture surfaces concomitant to the five materials studied: (a) UCT (b) CCT, (c) CVS, (d) CEC, (e) CC.

4. Discussion

Glass ceramics (GC) must be properly crystallized according to the manufacturer's instructions [5,46]. In the case of GC, an inadequate crystallization process may not only negatively influence their mechanical and optical properties [24,47], but also have a negative effect on their mechanical and optical properties [33,48] and increase the marginal gap as well [49]. Considering these aspects, this study aimed to compare the influence of heat treatment on the FR of different ceramic materials used in CAD/CAM systems.

The control group consisted of CCT that, upon receiving AHT following the manufacturer's indications, optimized its crystalline structure, improving its mechanical properties due to the high content of submicron particles of 0.5 μm , ~40% lithium disilicate crystals, 5% lithium phosphate, and finally 5% of small lithium aluminum silicate crystals interlocked in a glassy matrix enriched with zirconium [16,18,50]. Moreover, among the analyzed properties of the studied materials was the compressive strength (CR), an important parameter involved in determining the mechanical behavior of brittle materials [50]. As mentioned before, based on the findings of this study, the null hypothesis was rejected as significant differences were found between the groups of ceramics studied ($p < 0.05$).

As shown in Table 4, the CCT samples had higher values of force (437.462 N) compared to UCT (343.212 N). These results are in agreement with those of several authors, e.g., Riquieri et al. [51], who characterized the microstructure and evaluated the mechanical properties of two GC before and after AHT; Celtra© Duo (CD) had a higher FR of 251.25 MPa after crystallization, illustrating that AHT develops a fine, dense, homogeneous, and polish-resistant microstructure in the given material. Other studies assure that polishing performed manually generates more compressive stresses, creating more significant microstructural defects in those ceramics that are not subjected to AHT [28,52,53].

On the other hand, CEC obtained a response to FR of 434,968 N, similar to that of CCT. These results simply continue confirming the conclusions of previous studies, demonstrating that GC receiving AHT (following the manufacturer's indications) show significantly improved FR [54,55]. Importantly, a change in protocol could alter the mechanical behavior of ceramics [56]. For example, the cooling step is a sensitive protocol that must be controlled because lithium disilicate ceramics have a monoclinic, orthorhombic crystalline structure and are therefore anisotropic [57]. The mechanical properties of these brittle materials depend on the residual stresses that can develop due to thermal stresses and phase transformation, which can cause distortion, decreasing the fracture toughness due to the crystallographic orientation of each grain [58]. Prolonged heat treatment (THP) also has a negative influence on this orientation. In fact, an *in vitro* study performed by Schweitzer [47] evaluated the influence of THP on the FR of two GC with lithium disilicate and lithium silicate (CEC and CD); the FR of CEC increased considerably (to 344.82 MPa), as it received higher temperature during crystallization. Further, AHT is of particular interest with regard to the final composition of GC. Both the crystalline $\text{Li}_2\text{Si}_2\text{O}_5$ and the residual amorphous phase are responsible for the mechanical properties and optical characteristics of the GC. In the CEREC material, Tessera™, in addition to the elongated configuration of $\text{Li}_2\text{Si}_2\text{O}_5$, the crystalline phase also contains virgillite crystals $\text{LiAlSi}_2\text{O}_6$ embedded in a zirconium-enriched glass matrix, conferring to the material a higher density and therefore high FR [59,60]. Material manufacturers report that the addition of zirconia crystals increases the strength of GC [61]. However, a previous study claimed the opposite, stating that there are no clinical advantages for zirconia-reinforced LiO-SiO_2 [62]. A possible explanation behind this impasse could be the percentage of zirconium: a less than 5% ZrO_2 content presents a 70% crystalline phase in the amorphous matrix, whereas a more than 10% ZrO_2 reinforcement reduces the crystalline phase to 40% [63–65], significantly affecting the mechanical properties of a given GC. These assertions are supported by this study as herein, CSC displayed the lowest force values of (389.677 N). Although CC yielded “higher” results regarding force (436.604 N), its variability was higher; for this reason, more studies are suggested to support these CC results. In another study that focused on analyzing the influence of AHT on the mechanical behavior of four types of CAD-CAM lithium

silicate GC, CEC and Rosetta[®] SM (RSM) showed higher FR than lithium silicate ceramics with zirconia; in addition, CEC presented a higher Weibull modulus, certifying that this ceramic is more homogeneous and dense [66]. Other authors evaluated and compared the mechanical properties of nine CAD/CAM materials: in their study, after receiving AHT, CEC displayed significantly higher values ($p < 0.001$), with a minimum of 1.69, maximum of 2.46, and a standard deviation of 2.18 [67].

The evaluation and correlation of FS with FR is revealed as a crucial parameter to determine the mechanical strength of brittle materials GC [28], as evidenced in previous studies. The results suggest that materials subjected to AHT might be suitable for PFP as supported by previous research. In contrast, those that have not undergone AHT might be suitable for unitary fixed prostheses (UFP), as supported in a study by Vichi et al. comparing the FS of 4 GC which showed that the highest CEC values were recorded (350 MPa) after receiving AHT [68]. This trend is confirmed in an analysis by Freitas et al. who evaluated the FS of three ceramic groups, highlighting that UCT exhibited the lowest values (215 MPa) [69]. Furthermore, the results of our research align with the findings of Keshmiri et al. who investigated CVS and obtained FS results (347 MPa) [70] very similar to those obtained in our study. Despite the introduction of virgillite crystals that “improve” translucency [71], a study by Mangla et al. shows the opposite: UCT obtained the lowest values of MT (24.677 ± 0.187) and HT (27.447 ± 0.820) when compared to CEC MT (29.366 ± 1.243) and HT (30.771 ± 0.912) [19].

Undoubtedly, one of the benefits of CAD/CAM technology is that it provides an avenue for the rapid production of dental restorations [38,72,73]. However, it also presents challenges inherent in the subtraction process, as milling the ceramic creates microstructural defects in the restorations [74]. In addition, preparation design, crown design, die type, cement space, type of cementing agent, and thickness of the restorations together influence the strength of a ceramic material [75], so they should be evaluated independently. Consequently, 1 mm thick crowns using CCT ceramics are lower, $1911.4 \pm (468.4 \text{ N})$, in contrast to those found in CEC ($2995.3 \pm 880.6 \text{ N}$), regardless of the type of cementation [60]. Hamza et al. examined the FR of different CAD/CAM ceramic materials after applying a self-adhesive cementation protocol. The results obtained were much higher for CVS ($1742.9 \pm 102.7 \text{ N}$) compared to CEC ($1565.2 \pm 89.7 \text{ N}$) [76]. For this reason, in this study, we chose not to perform cementation in order to obtain precise and specific information exclusively on the mechanical behavior of CAD/CAM ceramics placed under thermal influence.

The modulus of elasticity provided by the die material is directly related to the mechanical strength of the material. In this regard, one study [76] evaluated the FR of ceramic crowns with respect to the modulus of elasticity of the die; it found that the fracture load increased significantly with increasing modulus of elasticity. Another study applied a compression test to 60 molar crowns composed of various types of CAD/CAM ceramics, cemented on epoxy resin dies, again showing a FR of 3100 N [77]. Several researchers agree with the argument that increasing the modulus of elasticity of the support material can increase the FR of a GC [78,79]. Although Yucel and Yondem [80] stated that ceramic crowns supported on stainless steel dies have higher FR values compared to ceramic crowns supported on dentin dies; they recommended the use of dies composed of materials with low elastic modulus for mechanical testing, simulating the clinical environment more accurately. Although a die material with these characteristics could have been chosen in this study, it is possible that breakage was anticipated earlier than that of the ceramic under study. Therefore, a cobalt–chromium metal support structure was selected in this study.

In the fractographic analysis, it was observed that once the critical stress value was reached, unstable cracks were generated, which facilitated crack propagation until failure occurred without the occurrence of plastic deformation, as expected with respect to ceramic materials. This finding is supported by previous studies [80,81]. In addition, Abad-Coronel et al. conducted a study that analyzed the fracture toughness of materials used in temporary

fixed prosthodontics. In their fractographic analysis, they found that acetal resin (AR) was the material that showed the highest percentage of deformation [82].

Considering the limitations of the methodology used in the present investigation and with the objective of obtaining the results of a complete biomechanical behavior of the material used for dental restoration, it is important to carry out future studies replicating the clinical conditions under which this study was performed. This is because thermal and chemical changes and humidity lead to the aging of the ceramic restoration outlined by CAD/CAM systems.

In the future, randomized controlled clinical studies are also needed to validate the biomechanical behaviors of CAD/CAM ceramic restorations, which can replicate various other clinical conditions.

Within the limitations of this study, the exclusive focus on fracture toughness through a compressive test, without addressing other properties such as flexural strength, hardness, and fracture toughness, stands out. In addition, the microstructure of the material was not examined since it was not the main objective of the research. Future studies are urged to incorporate microstructural analyses of ceramics, providing a more complete understanding of changes in the microstructure of ceramic materials after undergoing additional heat treatments.

5. Conclusions

In summary, AHT proves to be crucial for improving the mechanical properties of the studied GC with respect to dental restoration:

- In CCT, AHT optimizes the microstructure thanks to its advanced composition and the formation of new virgillite crystals, consequently increasing its FR, thus allowing for it to be resistant to possible fissures and cracks created by subtractive processes during the fabrication of restorations.
- The results given by CE allow for us to conclude that these ceramics have a high capacity of absorbing and distributing stresses without suffering fractures.
- Although CC displays the highest FR values, its high dispersion indicates inconsistencies; one must exercise caution while using it.

Importantly, it is essential to use a crystallization temperature that is suitable with regard to the size of the prosthesis, following the manufacturer's indications, in order to prevent the incidence of alterations in its mechanical properties.

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