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Effect of multiple firings on the physical properties of a CAD/CAM produced lithium disilicate restorative material

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Abstract

Objective: To evaluate the effect of multiple firing cycles on the mechanical properties and microstructure of a lithium disilicate CAD ceramic material.

Methods: Ceramic disc specimens (12 x 1.2 mm) were manufactured using lithium disilicate (IPS e.max CAD, Ivoclar, Leichtenstein). The discs were wet-ground with silicon carbide paper, sintered according to the manufacturer's instructions, then divided into five groups. Each group underwent a different number of firing cycles. Biaxial flexural strength tests were performed according to ISO 6872(2008)E. Fracture toughness and hardness were measured using the indentation strength method. XRD diffraction was used to generate information on the crystalline phase content by using a diffractometer and computer software, and colour difference was measured using spectrophotometry. Data was analysed using ANOVA and a two-tailed Students t-test ($p = 0.05$).

Results: Statistical analysis of the results with one-way ANOVA showed no significant difference ($p < 0.05$) in the mean biaxial flexural strength or fracture toughness between any of the groups tested. XRD analysis showed no noticeable crystalline phase change, nor were there any new crystallites in any of the test groups. Statistical analysis using a two-tailed Students t-test indicated a statistically significant ($p < 0.05$) and noticeable increase in the yellow chroma of the specimens with increased numbers of thermal cycles.

Conclusion. Thermal cycling of IPS e.max CAD appears to have no negative effect on strength or toughness using the protocols presented in this paper, however the chroma may be negatively affected with increased firings.

Introduction

The use of chair-side CAD/CAM (computer aided design/computer aided manufacturing) systems are increasing in popularity among dentists in practice. This is because of the improved accuracy achieved with this technology and the convenience of milling restorations at the chair-side when compared to the earlier available technologies (Nakamura et al., 2003, Frankenberger et al., 2011, Anadioti et al., 2014, Shembesh et al., 2016). A popular material used in the production of all-ceramic restorations is a lithium disilicate-based ceramic, IPS e.max CAD (Ivoclar, Leichtenstein) (Fasbinder et al., 2010, Tysowsky, 2012, Ivoclar, 2013, Ivoclar, 2014). The manufacturing process of this particular product involves a two-stage

crystallization process in which the lithium meta-silicate crystals are activated during the first processing step. This results in a "blue block" which demonstrates excellent processing properties (Apel et al., 2008). After the milling process is completed, a final sintering step dissolves the meta-silicate phase completely while the lithium disilicate crystallizes (Holand et al., 2006, Apel et al., 2008, Ivoclar 2009, Culp and McLaren 2010, Chaiyabutr 2011). During the manufacturing process of the prosthesis, the restoration may be thermal cycled as many as 4 to 5 times depending on the aesthetic requirements and the skill of the clinician. Kang et al. (2013) studied the effect of heat treatment on the biaxial flexural strength of two lithium disilicate CAD/CAM materials. This study however, focused on the crystalline firing cycles only, and found no statistical difference in flexural strength when comparing the specimens before and after the heat treatment (Kang et al., 2013). Aurelio et al. (2015) investigated the effect of extended glaze firing treatment on IPS e.max CAD. Their results showed an improvement in fracture strength, decreased surface roughness, and stability of the crystalline structure of the material. However, glaze cycles (manufacturer-recommended or modified by slow cooling) and firing below the glass transition phase, significantly reduced fracture strength (Aurelio 2015). More recently, Jalali et al. (2016) reported on the effect of multiple firing cycles on the micro tensile bond strength of lithium disilicate reinforced ceramics.

To date, no publication on the stability of the physical properties of IPS e.max CAD when subjected to multiple firing cycles can be found. Therefore, the purpose of this study was to determine the effect of repeated firing cycles on the biaxial flexural strength, fracture toughness, hardness, crystallography, and colour stability of the IPS e.max CAD restorative material.

Materials and methods

Specimen Preparation:

Lithium meta-silicate IPS e.max CAD (Ivoclar, Leichtenstein) low translucency ceramic milling blocks were manually precision turned on a lathe to 13 mm diameter cylinders, using a tungsten carbide blade. These were used to section 101 discs with a pre-crystallization thickness of 1.4 mm (Accutom 50, Struers,

Rodovre, Denmark). Prior to crystallization, the discs were wet ground on a lapping machine, then polished (Tegrapol 21, Struers) to 1 µm, using grit size FEPA 4000 silicon carbide abrasive paper (Struers). All but one of the discs were crystallised according to manufacturer’s instructions. This process dissolved the meta-silicate phase completely while the lithium disilicate crystallized (Ivoclar 2009). The discs were divided into different groups, each designated to a different test (Table 1).

Each group underwent a different number of corrective firing cycles, with each test conducted having their test groups referred to as outlined in Table 2. The firing cycles were carried out in accordance with the manufacturer’s firing parameters, as set out in Figure 1.

Biaxial flexural strength (piston on three balls)

Specimens (75) were divided into five groups (n=15) as outlined in Table 2. ISO standard 6872:2008(E) was followed to determine the biaxial flexural strength (piston on three balls) of the prepared ceramic discs (ISO 2008).

The discs were brought to failure using a universal testing machine (Instron, model 3369, Instron Corp, USA) with a cross-head speed of 1 mm/min. The maximum load at failure (F_{max}) was recorded using Instron Bluehill 3 software.

The biaxial flexural strength was calculated using the following formula: (ISO 2008)

Equation 1

$$\sigma = - 0.2387 P(X - Y) / b^2$$

Where:

- σ = Maximum centre tensile stress (MPa)
- P = Maximum load (N)
- b = Specimen thickness at fracture origin (mm)

And:

$$X = (1 + \nu) \ln(r_2/r_3)^2 + [(1 - \nu)/2](r_2/r_3)^2$$

$$Y = (1 + \nu)[1 + \ln(r_1/r_3)^2] + (1 - \nu)(r_1/r_3)^2$$

Where:

- ν = Poisson’s ratio
- r₁ = Radius of support circle (mm)
- r₂ = Radius of loaded area (mm)
- r₃ = Radius of specimen (mm)

Data were statistically compared with one-way analysis of variance (ANOVA) (p < 0.05) using Excel (Microsoft, Washington, USA).

Table 1. Test groups and amount of specimens prepared for each of the experimental procedures.

Test conducted	Specimens per group (n)
Biaxial Flexural strength	75 specimens divided into 5 groups (n=15)
Indentation fracture toughness	15 specimens divided into 5 groups (n=3)
Colour analysis	5 A2LT specimens divided into 5 groups (n=1)
X-ray diffraction	*6 specimens divided into 5 groups (n=1)

* The remaining specimen was kept in the lithium meta-silicate phase in order to establish a base XRD trace

Table 2. Experimental groups with the type and amount of firing cycles performed.

Experimental Groups	Type and number of firing cycles	
	Crystallization	Corrective firing
ClDi	1	-
F1	1	1
F3	1	3
F5	1	5
F7	1	7

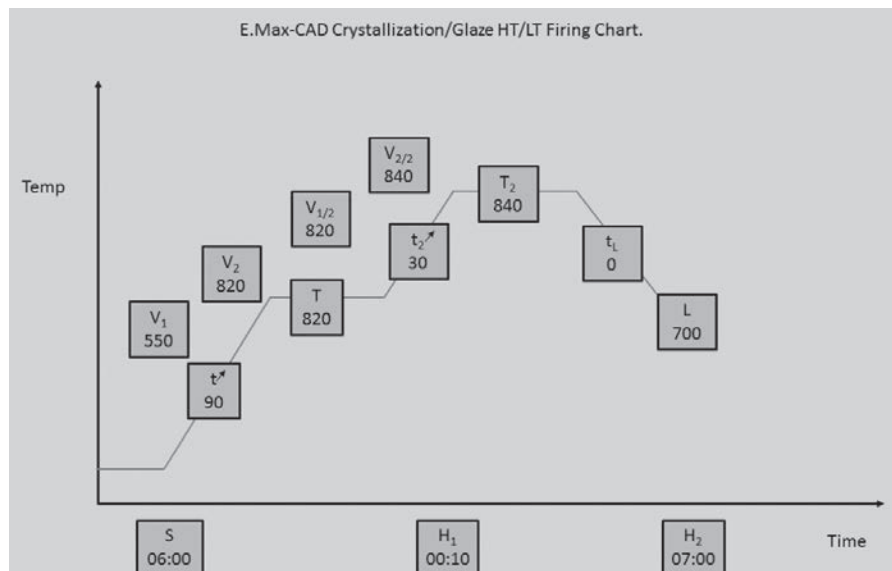


Figure 1. Prescribed firing parameters used during specimen preparation for the crystallization and corrective firing cycles for e.max CAD low translucency (LT), medium translucency (MT) and high translucency (HT) coloured milling blocks.

Fracture toughness:

Fracture toughness was determined using the indentation fracture toughness method, following the standard practices outlined in ASTM designation E2546-07 (Anstis et al., 1981, ASTM 2007).¹⁵ Specimens were divided into five groups (n=3). Using a Quorum K575X sputter coater (Quorum Technologies Ltd, Kent, UK), specimens were coated with a 5 µm gold-palladium layer to facilitate easy visualisation and accurate measurements of the cracks made by the indenter (Jung et al., 1999; Rhee et al., 2001; Albakry et al., 2003).

Five indents per specimen were made by applying a 50N load for 10 seconds using the universal testing machine mounted with a Vickers diamond pyramid indenter. Each indent and radial was measured and photographed within 30 seconds of indentation, using a 10X objective on a phase contrast microscope (Nikon YS2-H, Nikon, USA), with a 10 micron linear graticule (Olympus, Tokyo, Japan) and an attached digital camera (Canon Powershot A640, Canon, Japan).

The photographed radial cracks were measured using an image processing program (Image J, CITY, Maryland, USA). Fracture toughness (K_{IC}) was calculated using the following equation (Anstis et al., 1981):

Equation 2

$$K_{IC} = \kappa \left(\frac{E}{H} \right)^{0.5} \frac{P}{C^{3/2}}$$

Where:

κ = a constant 0.016

P = peak indentation load

c = combined lengths of the median/radial cracks from the centre of indent

H = hardness, obtained by the dividing P by the measured indent width squared

E = elastic modulus of the ceramic

Data were statistically compared as before.

Hardness test:

All the specimens used for the indentation fracture toughness testing were used to calculate the hardness. This was determined by dividing the force of indentation (P) by the area of indent (Anstis et al., 1981).

Equation 3

$$H = P/\alpha_0 a^2$$

Where

P = peak load

α_0 = numerical constant taken as 2

a^2 = impression half-diagonal

Data were statistically compared with one-way analysis of variance (ANOVA) ($p < 0.05$).

X-ray diffraction characterisation:

Six specimens were used for X-ray diffraction characterisation (XRD), one specimen for each of the five groups as outlined in Table 2. Control specimens

of lithium meta-silicate crystallite in the blue phase (designated, CMeta) and lithium disilicate (designated CLiDi) were used to produce characteristic XRD signatures. X-ray diffraction (X'Pert PRO, PANalytical, Almelo, The Netherlands) was carried out to determine if crystalline growth or phase changes occurred in the test specimens. Scanning electron microscopy (SEM) was used to detect changes in crystal sizes. Data were statistically compared using one-way analysis of variance (ANOVA) and a two-tailed unpaired heteroscedaisic Student's t test ($p < 0.05$) using Excel and StatPlus (AnalystSoft Inc, Virginia, USA) software.

Colour analysis:

For the colour stability analysis, a commonly used shade A2LT was used for specimen preparation following the same format as shown in Table 2. Five specimens were prepared as shown in Table 1. Each colour measurement was performed on a standardised black background after each firing cycle was completed, using a spectrophotometer (Vita Easys shade Compact; Vita, Germany) (Chu et al., 2010). Calibration of the spectrophotometer was performed after each recording. Measurements were recorded using the Commission Internationale de l'Eclairage CIE variables $L^*a^*b^*$. The total colour difference was calculated using the following equation (Gonuldas et al., 2014):

Equation 4

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}$$

Where ΔL ; Δa and Δb are the differences in the CIE colour-space parameters of the two shades.

A paired-samples t-test (Microsoft Excel) was performed to compare the colour difference between the groups.

Results

One-way ANOVA ($p < 0.05$) showed no significant difference in the mean biaxial flexure strength, fracture toughness or hardness between any of the groups tested, shown in Table 3.

Table 3. Flexural strength(Mpa), Fracture toughness (K_{IC}) and hardness (GPa) results for LiDi fired once for crystallization, followed by corrective firing cycles 1,3,5 and 7 times respectively (standard deviation in parenthesis)

Firing protocol	Mean Biaxial flexure strength (MPa)	Mean KIC (MPa.m0.5)	Mean Vickers Hardness (GPa)
CLiDi	451.0 (57)	1.61 (-0.08)	6.67 (0.33)
F1	389.5 (82)	1.62 (-0.09)	6.61 (0.30)
F3	422.1 (125)	1.64 (-0.08)	6.62 (0.46)
F5	417.47 (108)	1.51 (-0.24)	5.94 (1.35)
F7	458.74 (106)	1.54 (-0.16)	6.28 (0.92)

Specimens of the lithium meta-silicate crystallite in the blue phase and of lithium disilicate were used as controls CMeta and CLiDi. These produced characteristic XRD signatures for lithium meta-silicate and lithium disilicate (Figures 2 & 3). The disilicate was powder diffraction files (PDF) characterised as 04-009-8780 (Figure 4).

Test specimens F1, F3, F5, & F7

Lithium disilicate in the form of IPS e.max CAD, under our experimental conditions of 840°C for 1,3,5, or 7

“correction” firing cycles, did not show any observable crystallite phase change, nor were there any new crystallites identified. Peak size and shape altered in a way that indicated possible crystallite phase increase (Figure 4) and crystallite growth (Figure 5). Since large crystallites give rise to sharp Bragg peaks, growth of crystallites was detected in our specimens when the width of the peaks, the Full Width at Half Maximum height (FWHM) data from the XRD trace, was observed to decrease by half, as seen in figure 6. If an increase in crystallinity and lessening of amorphous content were to

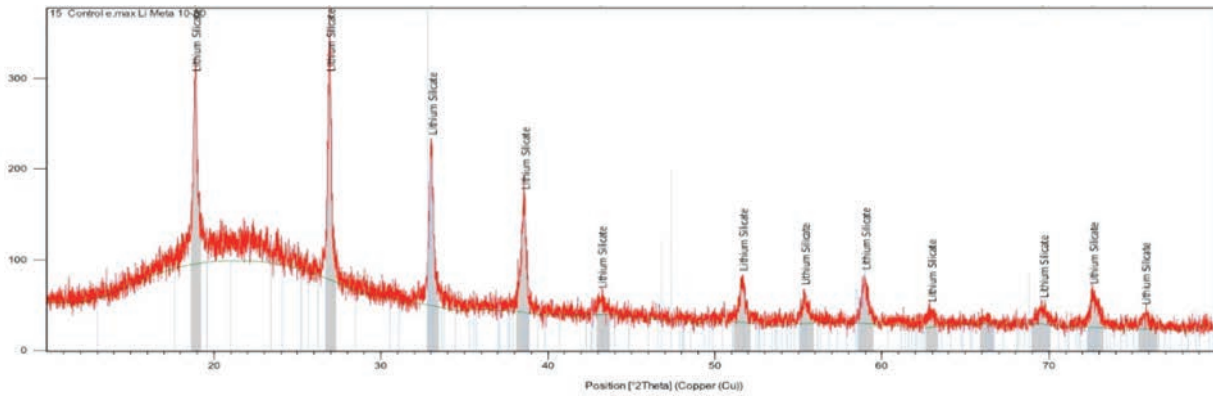


Figure 2. Control lithium disilicate CLiDi XRD trace 0-120° showing main peaks in the 10- 80 [°2Th.] range.

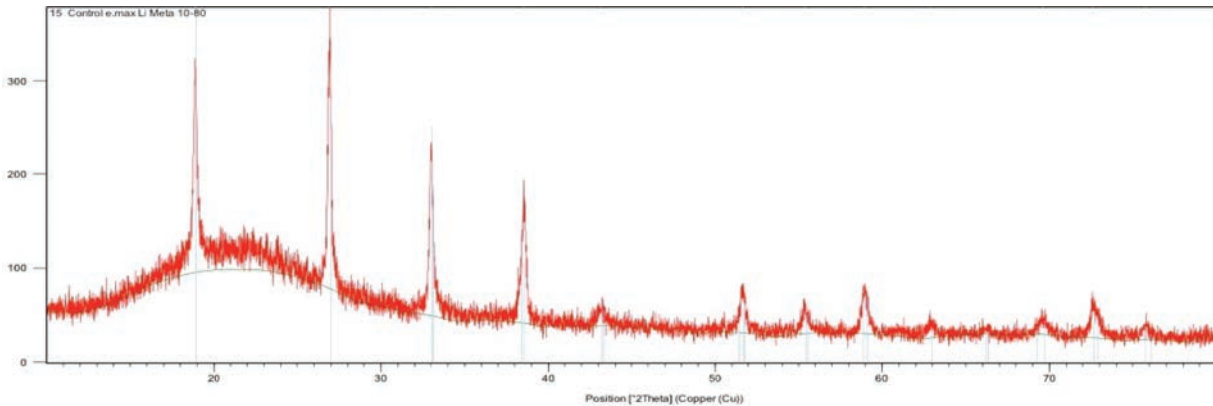


Figure 3. Control CMeta XRD 0-80° trace.

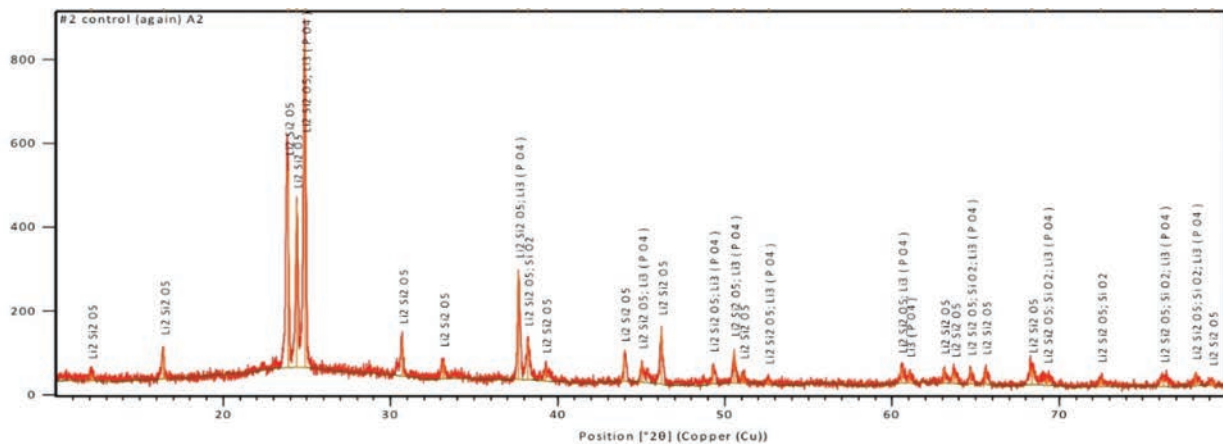


Figure 4. Lithium disilicate CLiDi trace showing 04-009-8780 and minor element 04-006-8566 peak matches.

occur, increased peak intensity would be observed, and this was able to be observed in the XRD trace as an increase in the height: background ratio of the Bragg peaks, as seen in Figure 5. This does not appear to be a large crystalline increase, however, but a rather tentative rise. This slight rise is not as convincing as the doubling of the FWHM data and may in fact be due to random variation.

Figure 7 shows all major peaks in F1 XRD trace (F1) are satisfied by lithium disilicate crystalline phases of either monoclinic PDF 04-009-8780 or orthorhombic PDF 01-082-2396. All major peaks in F3 XRD trace (F3) are satisfied by lithium disilicate crystalline phases of monoclinic PDF 04-009-8780. A minor amount of lithium orthophosphate was detected as trillithium phosphate (V).

This was PDF characterised by 04-006-8566. All major peaks in F5 and F7 XRD trace (F5 and F7) are satisfied by lithium disilicate crystalline phases of monoclinic PDF 04-009-8780 or orthorhombic PDF 01-082-2396. A minor amount of lithium orthophosphate was detected as trillithium phosphate (V). This was PDF characterised by 04-006-8566.

The colour change analysis indicated a statistically significant difference ($p < 0.05$) as well as a clinically observable ($CIE L^*a^*b^* E > 3$) darkening of the specimens, with increased numbers of firing cycles (Figures 8 and 9). This was very apparent after five firing cycles. The results also showed a visible colour shift when the IPS e.max CAD (A2 LT) material was thermally cycled according to the protocol followed in this study.

Height vs background ra=0 analysis

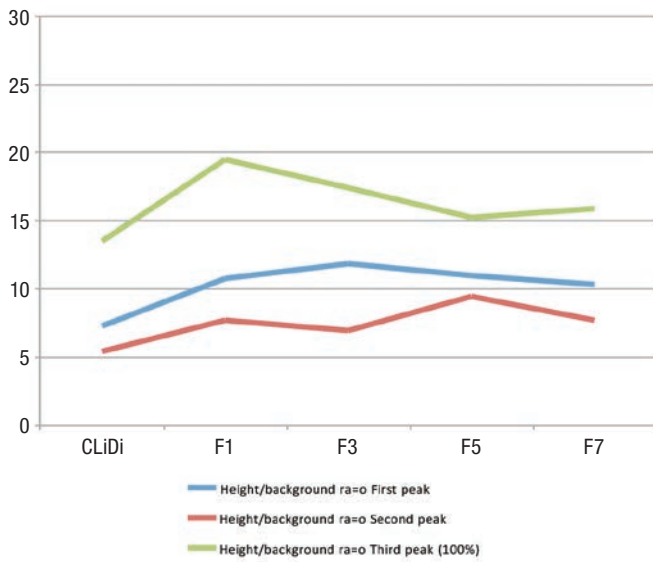


Figure 5. Height vs background ratio peak analysis.

Height/background ra=0 First peak

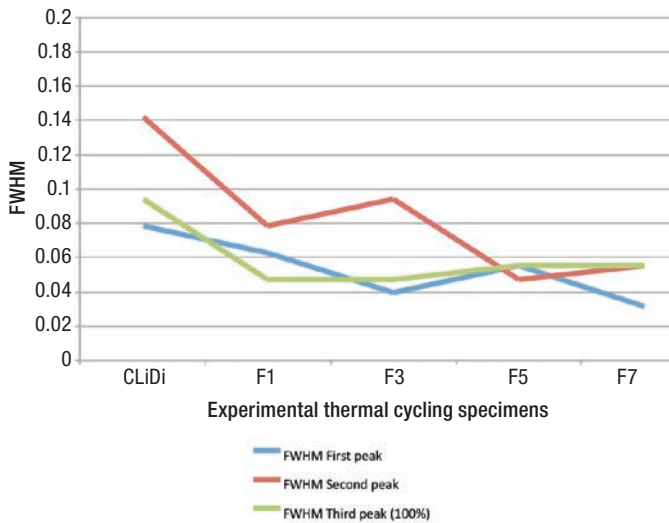


Figure 6. Full width at half maximum height analysis (FWHM).

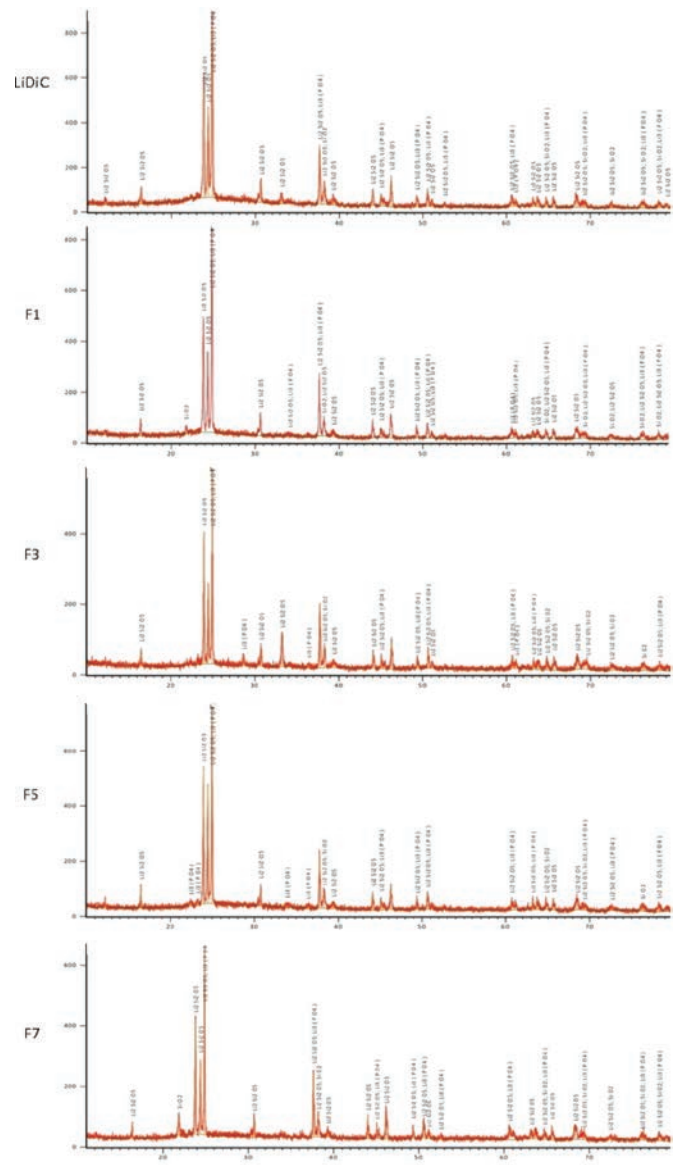


Figure 7. Comparison between the control and specimens F1, F3, F5, F7 XRD 10-80° traces showing 04-009-8780 and 04-006-8566 peak matches.

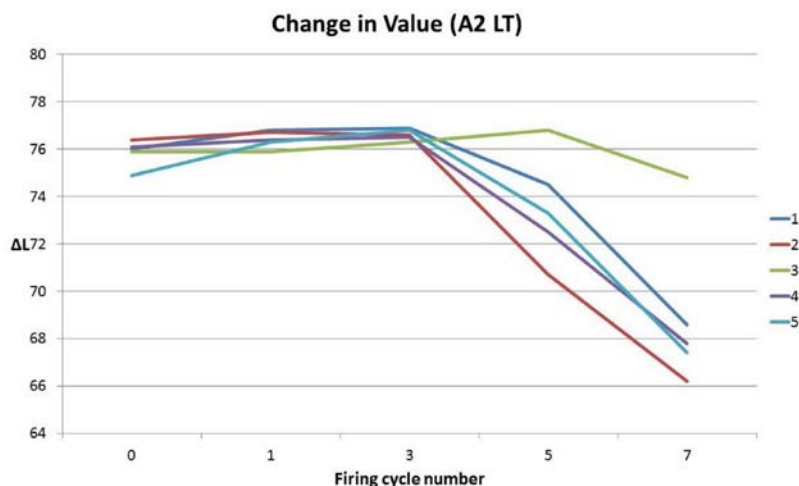


Figure 8. Variability of 5 A2LT IPS e.max CAD specimens (101 – 105) during thermal cycling, showing the change in CIE L*; where light is ranked towards 100 and dark towards 0.

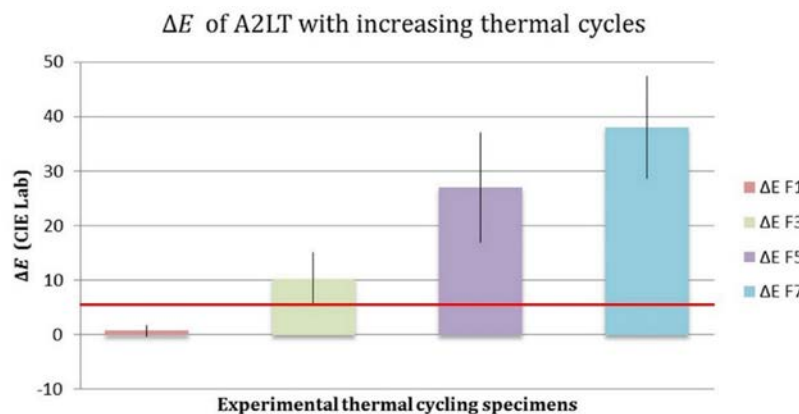


Figure 9. Showing the increasing amount of change in the A2LT ceramic with increased thermal cycles compared to what is clinically detectable the human eye (indicated by the red line). Where F1 have been fired once and F7, seven times.

Table 4. Paired t-test results showing significant difference ($p < 0.05$) between control and F7 groups of e.max CAD A2 LT material when subjected to multiple corrective firings.

Control vs F7	ΔE A2 LT e.max CAD
CIE L* variable	0.0108
CIE a* variable	0.0039
CIE b* variable	0.0005

This resulted in a darkening of the prosthesis ($p < 0.05$) as the red ($p < 0.01$) and yellow ($p < 0.01$) chroma increased, as indicated in Table 4.

Discussion

The purpose of this study was to determine the effect of repeated firing cycles on the biaxial flexural strength, fracture toughness, hardness, crystallography, and colour stability of IPS e.max CAD ceramic. The results for this study showed no statistically significant difference ($p < 0.05$) in mean strength, fracture toughness or the hardness values following thermal cycling from one to seven corrective firing cycles.

Ordinarily, glass ceramics are made by creating specific base glasses, which is done via a melting process. Controlled heating protocols nucleate and grow crystals within the glassy matrix (Höland et al., 2003). Multiple glass and crystalline phases may be present in a glass ceramic material. Lithium disilicate adds to the physical properties of glass ceramics, making it a strong and durable material (Alkadi and Ruse 2016). According to Holand and Beall (2012) the typical contents of lithium-disilicate glass ceramic include: silicon dioxide (SiO_2) and lithium oxide (Li_2O), which are responsible for the main crystal phase formation. A nucleating agent, such as di-phosphorous pentoxide (P_2O_5) and the glass matrix components, which can be either; chkalovite glass, potassium feldspar glass or alkaline earth feldspar glass. These three glass matrices can contain a favourable excess of SiO_2 of approximately 10%. This excess SiO_2 allows crystallite nucleation to occur. The specific amorphous glass constitution determines which heat treatment protocol is ideal for the base glasses during crystal nucleation (Holand 2012). The base crystalline phase of meta-silicate can quickly transform into lithium disilicate given a specific increase in temperature. The mechanism of lithium disilicate crystallisation due to various thermal treatments is core to this research. Borom et al. (1975) found that the relationship between strength and the crystalline phases present in lithium disilicate ceramics, was demonstrated as a function of the crystallization temperature (Borom et al., 1975). However, under the current experimental conditions, it appears that the lithium disilicate systems manufactured for use with CAD/CAM, are relatively resistant to thermally-induced crystalline X-ray diffraction detectable changes. No evidence of phase

change was expected, though given that crystallite phase change, i.e. melting of lithium disilicate, occurs at higher temperatures (in the range of 950°C) than used during this study (Holand et al., 2006). It is, however, likely that the glass phase is not completely depleted of crystallisable material, and crystalline growth could still occur. Indeed crystallite growth of a similar system had also been observed in two separate studies, both of which used SEM to quantify their finding (Oh et al., 2000, Albakry et al., 2004).

When considering the progressively wider peak intensities shown in the Full Width at Half Maximum Height (FWHM) data in Figure 6, the values of each of these three main peaks have halved, indicating definite crystal growth (Langford and Wilson 1978). This is attributed to processes of normal crystal growth or due to Ostwald ripening by virtue of increased molecular excitation and viscous relaxation effects (Brailsford and Wynblatt 1979, Marqusee 1984, Voorhees 1985). In our specimens, XRD analysis has been sufficient to show a change, however SEM may be a more successful tool to quantify crystalline growth. The observed change however, was not enough to influence the recorded strength, fracture toughness and hardness values in the present study.

The colour change results indicate that there is a discernable colour shift when IPS e.max CAD LT A2 ingots are thermally cycled according to the protocol followed in this study. This resulted in a darkening of the restoration ($p < 0.05$) as the red ($p < 0.01$) and yellow ($p < 0.01$) chroma increases. As Douglas et al. (2007) reported, if a E of 5.5 or greater exists then 50% of patients will ask for the restoration to be redone. In the present study, an average change in colour of 37.97 for A2 LT IPS e.max CAD demonstrated a clinically significant difference. Similar effects have been noted in IPS e.max Press (Ozturk et al. 2008) making this material appear darker and more reddish and yellow. The observed colour change of the lithium disilicate

in this study could be due to metal oxide colourant instability or crystalline growth and consequent alteration of light refraction. The increased chroma in the red and yellow hues can be attributed to the observations reported by two separate studies who reported that yellow/orange colourants were the least stable at manufacturers' recommended firing temperatures (Crispin et al., 1991, Lund and Piotrowski 1992). The noted increase in crystal size may also affect the matching of the optics of the glass and crystal phases, resulting in increased opacity of the material (Beall 1992, Höland et al. 2009, Holand et al. 2012). According to Holand and Beall (2012), the best translucency of a lithium disilicate ceramic is achieved when the glass matrix refractive index is adjusted to that of the lithium disilicate crystal. Therefore an increase in the crystalline dimension is likely to increase the opacity, perceived as decreased brightness, thus increasing the chroma of the existing colourants, which are in the yellow and red range (Holand et al., 2012).

Conclusion

Within the limitations of this study, the authors found:

- Repeated thermal firings had no statistically significant effect on the strength, fracture toughness or hardness on IPS e.max CAD lithium disilicate ceramic.
- There appears to be evidence on the XRD trace of crystalline growth but no phase changes were identified after these thermal treatments. Quantification of crystalline growth may be best analysed under SEM imaging of polished and appropriately etched surfaces.
- IPS e.max CAD A2LT material became noticeably darker and also more chromatic in the red and the yellow spectra of the CIE L*a*b* colour matrix after five thermal cycles.
- Future study in this field can include the effect of aging prior to mechanical property testing.

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