UNIVERSIDADE FEDERAL DE SANTA MARIA
CENTRO DE CIÊNCIAS DA SAÚDE
PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS ODONTOLÓGICAS

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QUEIMA ESTENDIDA DE GLAZEAMENTO EM CERÂMICAS DE CORTE DURO: CICATRIZAÇÃO DE DEFEITOS E EFEITO SOBRE AS PROPRIEDADES FÍSICO-MECÂNICAS

Santa Maria, RS
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Tese apresentada ao Curso de Doutorado do Programa de Pós-Graduação em Ciências Odontológicas, Área de Concentração em Odontologia, ênfase em Prótese Dentária, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Doutora em Ciências Odontológicas**.

Orientadora: Profa. Dra. Liliana Gressler May

Santa Maria, RS
2017

Ficha catalográfica elaborada através do Programa de Geração Automática
da Biblioteca Central da UFSM, com os dados fornecidos pelo(a) autor(a).

Aurélio, Iana Lamadrid
QUEIMA ESTENDIDA DE GLAZEAMENTO EM CERÂMICAS DE CORTE DURO: CICATRIZAÇÃO DE DEFEITOS E EFEITO SOBRE AS PROPRIEDADES FÍSICO-MECÂNICAS / Iana Lamadrid Aurélio.- 2017.
119 p.; 30 cm

Orientadora: Liliana Gressler May
Tese (doutorado) - Universidade Federal de Santa Maria, Centro de Ciências da Saúde, Programa de Pós-Graduação em Ciências Odontológicas, RS, 2017

1. CAD-CAM 2. Cerâmica Vítrea 3. Tratamento Térmico 4. Modificação de Defeitos 5. Resistência à Fadiga I. May, Liliana Gressler II. Título.

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Aprovado em 20 de outubro de 2017:

Liliana Gressler May, Dra. (UFSM)
(Presidente da Banca/Orientadora)

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Sara Fraga, Dra. (UNIFRA)

Viníejus Felipe Wandscher, Dr. (UNIFRA)

Rodrigo Othávio de Assunção e Souza, Dr. (UFRN) - Parecer

Santa Maria, RS
2017

DEDICATÓRIA

Com carinho,
aos meus pais João Luiz e Helenara, à minha avó Sonia, ao meu noivo Rodrigo
e à Professora Liliana.

AGRADECIMENTOS

E em meio à tantas incertezas, minha única certeza é a de que tenho muito a agradecer...

...Agradecer à Deus, ao nosso Senhor Jesus Cristo, e aos meus Guias de luz pela vigília constante. Os Senhores não evitam minhas quedas, mas me banham com força e fé para que eu aprenda a cair e depois levantar. Obrigado por tolerarem minhas imperfeições e meus dias de pessimismo e descrença. Obrigado pela família que tenho, e pelos tantos "irmãos" que ganhei, mesmo sendo filha única. Obrigado pelo amor e amparo incondicionais. Obrigado por me permitirem evoluir.

...Agradecer aos meus pais por me amarem infinitamente e por confiarem em mim a ponto de me permitir, agora aos 30 anos, ser também um pouco "mãe e pai" de vocês. Obrigado por priorizarem a minha formação, mesmo sabendo que isto implicaria em nos distanciarmos fisicamente de maneira precoce. Cheguei até aqui GRAÇAS À vocês! Cheguei até aqui POR vocês! Cheguei até aqui COM vocês!

...Agradecer à minha avó (mãe) Sonia pelo auto sacrifício exercido sem medir esforços, direcionado à tua família. Obrigado pela tua empatia conosco e com teu semelhante. Obrigado pela tua tolerância com aqueles que ainda não chegaram aos 91 anos, e que tanto tem a aprender contigo. Somos pra sempre um único coração!

...Agradecer ao meu noivo Rodrigo por me fortalecer e por me permitir fortalecê-lo. Obrigado por me tornar alguém melhor e por me permitir torna-lo melhor. Obrigado por acreditar que somos mais "prós do que contras". Obrigado por escolher viver ao meu lado e deixar tudo com mais sentido. Obrigado por me fazer gigante quando me sinto pequena. Obrigado por ser escudo e não barreira. Obrigado por ser só amor e não cobrança.

...Agradecer à minha orientadora Professor Liliana G. May por dar tanto sentido à palavra orientação. Me orientastes em todos os passos: naqueles que solicitei e principalmente em tantos outros que me guiastes mesmo sem eu pedir. Ensinamentos que absorvi com nosso convívio e que são de imenso valor. És fonte de amor, humildade, honestidade e competência. Obrigado pelo bom trato comigo e com os demais. Obrigado por ser calmaria e não tempestade.

Mãe, Pai, Vó, Rodrigo e Profa. Liliana, se me pedissem para escolher, os escolheria mil vezes! Obrigado por tudo! Amo vocês!

...Agradecer aos professores do Programa de Pós-Graduação em Ciências Odontológicas da Universidade Federal de Santa Maria pelos aprendizados transmitidos e por alimentarem diariamente o meu amor pela docência. Muito obrigado também aos professores que tive durante a graduação. Muito obrigado aos professores externos ao Programa e aos professores que compuseram as bancas examinadoras que fui submetida. Por onde eu caminhar, levarei comigo os seus exemplos. Por onde eu ensinar, levarei comigo as suas palavras. Por toda minha vida, levarei comigo as nossas estórias. Vocês foram, são, e continuarão sendo essenciais! Muito obrigado!

...Agradecer, sem exceção, aos meus colegas de caminhada ao longo da Pós-Graduação, sejam eles internos ou externos ao Programa. Obrigado pelo conhecimento trocado, por me fazerem sentir especial, pela risada frouxa das minhas palhaçadas. Obrigado pela torcida e

pelo companheirismo. Obrigado por serem remédio e não veneno. Que os nossos caminhos permaneçam entrelaçados e que todos vocês sejam surpreendidos por muitas conquistas, pois merecem o melhor!

...Agradecer em especial aos meus colegas Ana Maria, Andressa, Camila Rodrigues, Camilla Tibúrcio, Carine, Catina, Gabriel, Graziela, Luís Felipe, Sara e Vinícius, que por muita sorte também são meus amigos, meus irmãos! Que privilégio!

...Agradecer aos funcionários da "antiga reitoria" que mantêm o nosso espaço organizado e funcional, dissipando felicidade e amor em meio ao trabalho. Meu muito obrigado em especial ao "pessoal da portaria" – Emerson, Júlia, Bruna e Taila –, que sempre me recepcionou com um bom chimarrão e um baita sorriso. Vocês são alegria, colo e muita camaradagem.

...Agradecer à Jéssica e à Simone ou "Simo" pelos cuidados e dedicação para com cada um de nós. Obrigado por executarem tantas tarefas com maestria e não medirem esforços para que a nossa prioridade seja estudar e pesquisar. Obrigado "Simo" pelo alimento oferecido para o corpo e para a alma. Obrigado Jéssica pelo ouvido, pelo abraço, pelas surpresas e presentes, e por ser sempre "sem problemas" e nunca "não vai dar". Jéssica e Simone, a "música" do Programa só toca graças à regência de pessoas como vocês.

...Agradecer o Centro Universitário Facvest pela confiança, me permitindo exercer a docência que tanto amo. Obrigado aos meus colegas professores pela recepção e por tornarem nosso ambiente de trabalho acolhedor.

...Agradecer os meus alunos pelas experiências compartilhadas, pelo respeito, por me valorizarem e por acreditarem em mim. Obrigado pelo carinho e preocupação mesmo quando estamos distantes. Feliz de mim que participo da formação de vocês e ainda os ganho como amigos. Obrigado por tornarem Lages tão legal! Obrigado por serem abrigo e não solidão.

...Agradecer à todos os amigos que não foram diretamente citados, mas que fizeram e fazem parte da minha trajetória, e que estão sempre torcendo por mim. Agradeço em especial às minhas amigas Bruna, Renata, Shalimar e Vivian pela preocupação e carinho constantes. Obrigado por compreender a minha ausência neste período. Vocês são mais que especiais.

...Agradecer à minha filha de quarto patas, Haru, que com muita facilidade me arranca sorrisos e que traz muita alegria à minha vida. Que teve seus passeios e suas brincadeiras muitas vezes sacrificados ao longo da minha formação. Obrigado, Deus, por me reservar a Haru e tantos outros cachorrinhos que estiveram junto de mim e que tornaram minha vida mais doce.

...Agradecer às oportunidades!!!

...Agradecer a cada um que me deu uma oportunidade; que todas elas retornem à vocês. Não há quem cresça sem a ajuda do outro. Quem divide, multiplica. A todos aqueles que me deram uma oportunidade, como profissional, como aluna, como amiga, meu muito obrigado! Obrigado por serem ponte e não corda bamba. Obrigado por serem chance e não egoísmo!

RESUMO

QUEIMA ESTENDIDA DE GLAZEAMENTO EM CERÂMICAS DE CORTE DURO: CICATRIZAÇÃO DE DEFEITOS E EFEITO SOBRE AS PROPRIEDADES FÍSICO-MECÂNICAS

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ORIENTADORA: Liliana Gressler May

Esta tese avaliou empregar a queima estendida de glazeamento (GE) como alternativa à queima convencional (G, recomendada pelo fabricante) para o acabamento de cerâmicas densamente sinterizadas para usinagem em sistemas CAD-CAM (*Computer Aided Design; Computer Aided Machining*). Primeiramente, investigou-se o efeito de ambas as queimas na cicatrização de defeitos, tensões residuais, características ópticas e na estrutura cristalina dos materiais. Espécimes retangulares (≈ 14 x 12 x 1,5mm), obtidos da secção de blocos usináveis de cerâmicas feldspática (FEL), leucítica (LEU), à base de dissilicato de lítio (DIS) e de silicato de lítio reforçada por zircônia (SLZ), foram divididos em grupos (n=5) conforme a queima aplicada: G, GE (15min à temperatura de manutenção, seguido de resfriamento lento) e C (nenhum/controle). Defeito gerado por indentação Vickers foi analisado em microscópio eletrônico antes e após a queima (n=1) para avaliação de sua cicatrização. As tensões residuais foram determinadas pela técnica da indentação. As alterações de cor (ΔE) e de translucidez (ΔRC) dos espécimes após a queima foram mensuradas, respectivamente, pelo método CIEDE2000 e pela razão de contraste. A estabilidade da microestrutura cristalina foi analisada por difração de raios-X (n=1). Independente do material, GE teve maior capacidade de cicatrizar defeitos e produziu tensões residuais compressivas, quando comparado ao G, que gerou tensões trativas. GE promoveu alterações de cor imperceptíveis nas cerâmicas FEL e LEU, perceptíveis/aceitáveis para DIS, e clinicamente inaceitáveis para SLZ. G não modificou perceptivelmente a cor dos materiais. Após GE, SLZ ficou perceptivelmente menos translúcida. A estrutura cristalina de todas as cerâmicas permaneceu estável após G e GE. Diante disso, em um segundo momento, buscou-se averiguar o efeito de GE e G sobre a resistência flexural à fadiga (RFF) de discos de LEU e DIS usinados (CEREC inLab MC XL). Os espécimes foram divididos por material nos grupos (n=20) G, GE e C e, submetidos à ensaio de fadiga pelo teste da escada, na presença de água (*piston-on-three ball*, 500.000 ciclos, 20Hz e carregamento sinusoidal). A rugosidade superficial dos discos foi mensurada em perfilômetro de contato, antes e após as queimas. Análise fractográfica foi executada para identificação da origem das falhas (n=1). As médias e desvio padrão da RFF dos grupos foram submetidos à análise de variância (ANOVA-1 fator) e teste de Tukey (α=0,05). Testes estatísticos para comparações pareadas dos dados de rugosidade foram selecionados em função da normalidade/homocedasticidade dos mesmos. Para ambos os materiais, GE alcançou a maior RFF, estatisticamente superior ao G e ao grupo controle, C. A rugosidade de LEU e DIS não foi alterada após as queimas G e GE. As marcas fractográficas levaram à identificação de defeitos originários da superfície de tração. Assim, conclui-se que a queima GE: conferiu maior selamento dos defeitos em relação ao G, desenvolveu tensões residuais toleráveis, sem alterar as propriedades ópticas além do limiar clínico e modificar a microestrutura das cerâmicas FEL, LEU e DIS; e, otimizou os valores de resistência flexural à fadiga para espécimes usinados de LEU e DIS, podendo vir a ser uma alternativa viável para acabamento desses materiais.

Palavras-chave: CAD-CAM. Carregamento Cíclico. Cerâmica Vítrea. Difratometria. Distribuição de Tensões. Estabilidade de Cor. Resistência à Fadiga. Selamento de Trincas. Tratamento Térmico.

ABSTRACT

EXTENDED GLAZE FIRING ON CERAMICS FOR HARD MACHINING: CRACK HEALING AND EFFECT ON THE PHYSICAL-MECHANICAL PROPERTIES

AUTHOR: Iana Lamadrid Aurélio
ADVISER: Liliana Gressler May

This thesis evaluated an extended glaze firing (EG) versus the conventional glaze firing (G, manufacturer-recommended) for finishing of densely sintered ceramics indicated for machining in CAD-CAM (Computer Aided Design; Computer Aided Machining) systems. Initially, the effects of both firings on ceramic surface crack healing, residual stresses, optical characteristics and crystalline structure were evaluated. Rectangular specimens (≈ 14 x 12 x 1,5mm) obtained from the section of feldspathic (FEL), leucite- (LEU), lithium disilicate- (DIS), and zirconia-reinforced lithium silicate-based (ZLS) prefabricated ceramic blocks were divided into groups according to the applied firing (n=5): G, EG (15min at dwell time, following slow cooling) and C (control/no heat treatment). Defect generated by Vickers indentation was analyzed in an electronic microscope before and after firing (n=1) to evaluate its healing process. Residual stresses were determined by indentation technique. Specimen color differences (ΔE) and variations in translucency (ΔCR) were measured after firing, respectively, by CIEDE2000 method and contrast ratio. Stability of crystalline microstructure was analyzed by X-ray diffraction (n=1). Regardless of the material, EG had greater ability to heal defects when compared to G and also produced compressive residual stresses, while G generated tensile stresses. Color differences after EG were imperceptible in FEL and LEU ceramics, perceptible/acceptable for DIS, and clinically unacceptable for ZLS. G did not perceptibly change the color of the materials. After EG, ZLS ceramic was perceptibly less translucent. Crystalline phase of all the ceramics remained stable after G and EG. Therefore, in a second moment, we intended to investigate the effect of EG and G firings on the flexural fatigue strength (FFS) of LEU and DIS ceramics. For this, ceramic blocks were machined in disc-shaped specimens using CEREC inLab MC XL and were divided into six experimental groups (n=20) according to the ceramic – LEU or DIS – and the firing – G, EG or C. Specimens were submitted to fatigue using staircase test design in water (piston-on-three ball, 500.000 cycles, 20Hz and sinusoidal loading). Surface roughness of the discs was measured using a contact profilometer, before and after firing. Fracture origin was analyzed by scanning electron microscopy (SEM). Means and the standard deviation of FFS were submitted to analysis of variance (ANOVA-1 factor) and Tukey's test ($\alpha = 0.05$). The most appropriate statistical tests for pared comparisons of the roughness data were selected based on normality/homoscedasticity results. For all the tested materials, EG achieved the highest FFS, statistically higher than G and C groups. The surface roughness of the LEU and DIS ceramics did not change after G and EG. Fractographic marks led to the identification of fracture originating from the tensile surface. Thus, it was concluded that the EG cycle promoted greater defect healing in relation to G, developed tolerable residual stresses, and did not alter neither the optical properties beyond the clinical threshold, nor the microstructure of the FEL, LEU and DIS ceramics; in addition, it have improved the flexural fatigue strength values for LEU and DIS machined specimens. Therefore, it seems to be a promising alternative for finishing such materials.

Keywords: CAD-CAM. Color Stability. Crack Healing. Cyclic Loading. Diffractometry. Fatigue Strength. Glass-ceramics. Heat Treatment. Stress Distribution. Stress Profile.

SUMÁRIO

1 INTRODUÇÃO

Os métodos tradicionais de confecção de restaurações cerâmicas são descritos como demorados e sensíveis às variáveis laboratoriais (LI; CHOW; MATINLINNA, 2014). Assim, a usinagem de restaurações dentárias em sistemas CAD-CAM (*Computer Aided Design/Computer Aided Machining*), a partir de blocos cerâmicos pré-fabricados obtidos por um processo controlado, padronizado e que resulta em uma cerâmica mais homogênea e com menores chances de incorporação de defeitos (BELLI et al., 2016; WENDLER et al., 2017) ganhou evidência ao longo das últimas duas décadas (REKOW et al., 2011). Dependendo do tipo de material, os blocos são disponibilizados parcial ou totalmente sinterizados. Quando blocos densamente sinterizados são utilizados, a usinagem é denominada "de corte duro". Cerâmicas feldspática, vítrea reforçada por leucita e à base de dissilicato de lítio em estágio pré-cristalizado são materiais comumente disponíveis para este tipo de usinagem (DENRY; HOLLOWAY, 2010). Recentemente, cerâmicas à base de silicato de lítio reforçadas por zircônia também foram desenvolvidas para tal fim (DENRY; KELLY, 2014).

As taxas de sucesso clínico para restaurações cerâmicas obtidas a partir da usinagem de blocos densamente sinterizados são em geral promissoras (>80%), muito embora a fratura do material cerâmico ainda apareça como a principal causa de falha/complicação técnica destas restaurações (GUESS et al., 2013; OTTO; SCHNEIDER, 2008; PJETURSSON et al., 2015, 2017; REICH et al., 2014; SAILER et al., 2015, 2016; REISS; WALTHER, 2000). Alguns estudos laboratoriais indicam que o processo de usinagem (KELLY et al., 1991; SCHERRER et al., 2011), ou que defeitos superficiais oriundos da face usinada sob tração (MORES et al., 2017; ZUCUNI et al, 2017), estejam associado às falhas de espécimes totalmente cerâmicos e à redução dos valores de resistência flexural (FRAGA et al., 2015; WANG; ABOUSHELIB; FEILZER, 2008) e de resistência à fadiga do material (FRAGA et al., 2017). De fato, a natureza friável da cerâmica representa um desafio para a usinagem (DENRY, 2013; MARSHALL et al., 1983), a qual promove danos superficiais e sub-superficiais no material (DENRY; HOLLOWAY; TARR, 1999), que por sua vez deveriam ser potencialmente minimizados por procedimentos indicados para a finalização (acabamento) das peças cerâmicas, como as queimas de glazeamento.

O glazeamento é indispensável aos procedimentos de caracterização extrínseca, aprimorando a aparência estética das restaurações monolíticas usinadas em cerâmica pura, que tendem a ser monocromáticas (SADOWSKY, 2006). Entretanto, mesmo que as queimas de glazeamento estejam por vezes associadas à aplicação de pigmentos (*stains*) e pastas ou

sprays de glaze e que, convencionalmente a adição destas películas à superfície cerâmica sejam estratégias para melhorar a resistência das restaurações (MORES et al., 2017), o processo de usinagem produz defeitos ao longo de todo o material, inclusive na face de cimentação, que sofre apenas a ação do tratamento térmico (CHEN et al., 2010). Resultados recentes demonstram que os ciclos de glazeamento não foram capazes de eliminar os defeitos causados pela usinagem (DENRY, 2013), ou até mesmo tiveram um efeito deletério (AURÉLIO et al., 2015; FRAGA et al., 2015) sobre a resistência mecânica de materiais vitrocerâmicos. Surge assim a necessidade de estudos com ciclos térmicos alternativos, na busca por um tratamento de finalização (acabamento) que otimize o desempenho de cerâmicas densamente sinterizadas para usinagem em sistemas CAD-CAM.

Neste intuito, Aurélio et al. (2015) adaptaram os preceitos do *annealing* [1] (recozimento) (PRESTON, 1925) a um protocolo experimental de queima estendida de glazeamento que fosse factível em laboratório. Os autores testaram prolongar por 15 minutos, com posterior resfriamento lento, o tempo de manutenção do ciclo de glazeamento recomendado pelo fabricante (1,5 minutos à 790ºC, seguido de abertura imediata do forno) (IVOCLAR VIVADENT, 2014) para uma vitrocerâmica leucítica. Aumento significativo nos valores de resistência flexural foi alcançado pelo grupo submetido à queima estendida, quando comparados aos espécimes que receberam a queima de glazeamento convencional. Hipoteticamente, o resultado foi creditado à possível capacidade da queima estendida de, ao menos, arredondar e/ou reduzir a profundidade dos defeitos deixados pela usinagem, associada a um estado de tensões residuais favoráveis desenvolvidos na superfície dos espécimes após o tratamento.

Determinados tratamentos térmicos demonstraram ser capazes de reduzir a viscosidade da matriz vítrea e, por capilaridade (GIRARD; FAIVRE; DESPETIS, 2011; HRMA; HAN; COOPER, 1988), diminuir a profundidade das fissuras, arredondar as pontas das trincas (FAIRHURST et al., 1992; GRIGGS; THOMPSON; ANUSAVICE, 1996) e, até mesmo, selar os defeitos (DENRY; HOLLOWAY; TARR, 1999; LENZ et al., 2002). Concomitantemente, estudos sugerem que materiais cerâmicos constituídos por silício (Si),

[1] Tratamento térmico que visa, numa primeira etapa, manter o material aquecido à uma temperatura compreendida entre a transição vítrea (T_g) e o ponto de amolecimento, de modo que a viscosidade e a difusão atômica sejam ideais para o alívio de tensões residuais em um intervalo de aproximadamente 15 minutos; e, em seguida, resfria-lo cuidadosamente (CALLISTER, 2012). Esse procedimento tem sido aplicado em cerâmicas odontológicas no intuito de promover o alívio de tensões oriundas do processamento (FISCHER et al., 2005) e cicatrização de defeitos (DENRY; HOLLOWAY; TARR, 1999).

quando expostos à tratamento térmico adequado, sofrem reação de oxidação e geram produtos viscosos à base de SiO_2 (cristalino ou amorfo) que tendem a selar os defeitos (GREIL, 2012; QUEMARD et al., 2007).

Alguns autores acreditam que a usinagem seria capaz de introduzir uma fina faixa de tensões compressivas na superfície da cerâmica, com efeito positivo sobre sua resistência (GARVIE; HANNINK; PASCOE, 1975; MARSHALL et al., 1983). Dessa forma, entende-se que talvez os ciclos térmicos aplicados às cerâmicas usinadas não devessem eliminar tais tensões, a fim de não reduzir a resistência do material (AURÉLIO et al., 2015; GIORDANO; CIMA; POBER, 1995).

Cabe ressaltar que as queimas idealmente não devem interferir no efeito desejado do glazeamento, nem sobre as características ópticas da restauração, haja vista a instabilidade de alguns componentes cerâmicos frente à temperatura (GONULDAS; YILMAZ; OZTURK, 2014; OZTURK et al., 2008; PIRES-DE-SOUZA et al., 2009; YILMAZ; GONULDAS; OZTURK, 2014). O tratamento térmico, dependendo da temperatura e do período de tempo aplicados, pode alterar a fração volumétrica das fases (MACKERT et al., 2003; MACKERT; EVANS, 1991, 1992; MACKERT; RUSSEL, 1996; YILMAZ; GONULDAS; OZTURK, 2014), modificar (BARREIRO; VICENTE, 1993) ou degradar (FRAGA et al., 2015) seus constituintes, determinando novas propriedades mecânicas, químicas e físicas para um dado material (CALLISTER; RETHWISCH, 2012). Desta maneira, se faz necessário que os estudos tragam informações a respeito do efeito dos tratamentos térmicos sobre as propriedades ópticas e estrutura cristalina do material.

Para o estudo da influência do tratamento térmico sobre as propriedades mecânicas das cerâmicas submetidas à usinagem, os ensaios de fadiga cíclica se apresentam de maneira complementar aos ensaios monotônicos tradicionais, pois reproduzem uma condição mais próxima do que ocorre clinicamente (MAY et al., 2015; WISKOTT; NICHOLLS; BELSER, 1995). Salienta-se que informações sobre o impacto das etapas envolvidas ao longo do processamento das restaurações, tal como a usinagem e o tratamento térmico, no comportamento cerâmico frente à carregamentos cíclicos são ainda bastante escassas na literatura, merecendo investigação.

Assim, considerando os contextos expostos, o presente trabalho de tese visou, primeiramente, avaliar o efeito das queimas estendida e convencional (recomendado pelo fabricante) de glazeamento na cicatrização de defeitos, tensões residuais, características ópticas e na estrutura cristalina de quatro cerâmicas de corte duro, com diferentes microestruturas: feldspática, reforçada por leucita, à base de dissilicato de lítio e à base de

silicato de lítio reforçada por zircônia. Em continuidade, e tendo como base os resultados obtidos nesta primeira etapa, almejou-se investigar o efeito das queimas estendida e convencional de glazeamento sobre a resistência flexural à fadiga de duas cerâmicas com diferentes microestruturas (reforçada por leucita e à base de dissilicato de lítio), em comparação à espécimes somente usinados. Avaliação da rugosidade superficial após as queimas e análise fractográfica para identificação da origem das falhas também foram executadas.

2 REVISÃO DE LITERATURA

2.1 CERÂMICAS INVESTIGADAS

A microestrutura, composição e indicações clínicas das cerâmicas investigadas neste trabalho de tese estão descritas em detalhe na Tabela 1. Todos os materiais são disponibilizados no formato de blocos pré-fabricados para usinagem automatizada considerada "de corte duro", executada por sistemas CAD-CAM.

Tabela 1 – Cerâmicas para usinagem de corte duro utilizadas neste estudo.

Cerâmica	**Microestrutura**	**Composição[a]**	**Indicações**
VITABLOCS MarkII (Vita, Zahnfabrik, Bad Säckingen, Alemanha)	Feldspática. Cristais da família dos feldspatos (sanidina - $KAlSi_3O_8$; nefelina - $NaAlSiO_4$; albita - $NaAlSi_3O_8$) <20% vol dispersos na matriz vítrea.	Al_2O_3; SiO_2; K_2O e Na_2O; CaO; TiO_2; outros óxidos.	Recomendado para: inlays, onlays. Possível para: laminados, coroas unitárias parciais e totais em região anterior e posterior, e *endocrowns*.
IPS Empress CAD (Ivoclar-Vivadent, Schaan, Liechtenstein)	Vítrea reforçada por leucita. Cristais de leucita ($K_2O.Al_2O_3.4SiO_2$) ≈35-45%vol dispersos na matriz vítrea.	Al_2O_3; SiO_2; K_2O; Na_2O; outros óxidos e pigmentos.	Inlays, onlays, laminados, coroas unitárias totais e parciais em região anterior e posterior.
IPS e.max CAD (*Monolithic Solutions*, Ivoclar-Vivadent, Schaan, Liechtenstein)	Vítrea à base de dissilicato de lítio. Quando totalmente cristalizada: cristais de dissilicato de lítio ($Li_2Si_2O_5$) ≈70%vol envoltos por matriz vítrea.	SiO_2; Li_2O; K_2O; P_2O_5; ZrO_2; ZnO; Al_2O_3; MgO e outros óxidos.	Laminados, inlays, onlays, coroas unitárias anteriores e posteriores e pontes fixas de até 3 elementos (até segundo pré-molar).
VITA SUPRINITY (Vita, Zahnfabrik, Bad Säckingen, Alemanha)	Vítrea à base de silicato de lítio reforçada por zircônia. Quando totalmente cristalizada: cristais de dissilicato de lítio ($Li_2Si_2O_5$) e metassilicato de lítio (Li_2SiO_3) (DENRY; KELLY, 2014) envoltos por matriz vítrea contendo zircônia (ZrO_2) ≈10%vol (KRÜGER et al., 2013).	SiO_2 ; Li_2O; K_2O; P_2O_5; ZrO_2; Al_2O_3; CeO_2 e pigmentos.	Inlays, onlays, laminados, coroas unitárias totais e parciais em região anterior e posterior. Restaurações unitárias sobre implantes em região anterior e posterior.

[a] Composição descrita de acordo com informações dos fabricantes

O maior teor vítreo presente nas cerâmicas feldspática e leucítica (Tabela 1) confere à esses materiais excelentes propriedades ópticas que mimetizam as características da estrutura dental. Em contrapartida, essa microestrutura predominantemente vítrea faz com que estas cerâmicas apresentem baixa resistência mecânica – em torno de 154 MPa para a fedspática à 160 MPa para a leucítica, segundo os fabricantes – quando comparadas às cerâmicas à base de dissilicato de lítio e de silicato de lítio reforçada por zircônia (360 e 420 MPa, respectivamente, de acordo com os fabricantes), que contam com maior agregado cristalino, mas, às custas disto, são menos translúcidas (KELLY, 2004, 2008).

Recentemente, Belli et al. (2016) apresentaram estimativas de vida promissoras para restaurações posteriores unitárias obtidas por meio de usinagem de corte duro, onde o tempo estimado para que 10% destas restaurações viessem a falhar foi de no mínimo 10,9 anos. Inlays confeccionas a partir das cerâmicas vítreas Vita Mark II e Dicor (Dentsply, Konstanz, Alemanha) alcançaram taxas de sobrevivência de 94,7% e 85,7% após 5 e 10 anos, respectivamente (ZIMMER et al., 2008). Coroas unitárias em região de molares da cerâmica Vita Mark II apresentaram sobrevida de 94,4% após 44.6 ± 13 meses (BINDL; MÖRMANN, 2004). Estudos clínicos com coroas unitárias de IPS e.max CAD reportaram a ausência de falhas em 93% e em 100% das restaurações ao final de 46 meses (REICH et al., 2014) e após dois anos de acompanhamento (FASBINDER et al., 2010), respectivamente. Coroas parciais de vitrocerâmica reforçada por leucita IPS Empress ProCAD (Ivoclar Vivadent, Liechtenstein) apresentaram 100% de sobrevivência durante um período de observação entre 1 e 4 anos (DENISSEN et al., 2002) enquanto que, coberturas parciais em molares alcançaram os 97% após 7 anos (GUESS et al., 2013). Atualmente, a cerâmica IPS Empress CAD (Ivoclar Vivadent, Liechtenstein) é a sucessora da IPS Empress ProCAD. Devido à recente introdução no mercado odontológico, ainda não existem dados publicados acerca do acompanhamento clínico de restaurações à base de silicato de lítio reforçado por zircônia.

Apesar das otimistas taxas de sucesso clínico relatadas para as restaurações cerâmicas usinadas a partir dos materiais selecionados neste estudo ou similares, cabe a ressalva que alguns dados são à curto prazo. Além disso, a fratura do material ainda se destaca como falha/complicação técnica destas restaurações (GUESS et al., 2013; OTTO; SCHNEIDER, 2008; PJETURSSON et al., 2015, 2017; REICH et al., 2014; SAILER et al., 2015, 2016; REISS; WALTHER, 2000). Isto, graças à característica de fragilidade ou “friabilidade” das cerâmicas, que indica que estas suportam pouca ou nenhuma deformação plástica antes de fraturar (ANUSAVICE, 1998). Dessa forma, pressupõe-se que processos que envolvam o desgaste do material, e invariavelmente danos na superfície, sub-superfície, e até mesmo ao

longo de todo o interior da cerâmica, tal como a usinagem (DENRY; HOLLOWAY; TARR, 1999), venham a exercer um impacto significativo sobre o seu desempenho (FRAGA et al., 2015, 2017; KELLY et al., 1991; SCHERRER et al., 2011); e que as fraturas se originem a partir de regiões com concentração de tensões trativas, por meio da propagação dos defeitos (CESAR et al., 2006; MAGNE et al., 1999; MORES et al., 2017; ZUCUNI et al, 2017).

2.2 IMPACTO DA USINAGEM NO COMPORTAMENTO MECÂNICO DOS MATERIAIS CERÂMICOS

Importante evolução tecnológica na área dos materiais dentários tem sido perceptível ao longo das últimas décadas, tendo os processos automatizados de produção ocupado um papel de destaque e contribuído significativamente para o desenvolvimento de uma odontologia restauradora com melhores padrões de segurança e confiabilidade (VAN NOORT, 2012).

A usinagem pode ser realizada a partir da fresagem de blocos cerâmicos densamente sinterizados, considerada "de corte duro" ("*hard machining*"), como é o caso das cerâmicas utilizadas neste estudo, ou ainda, utilizando-se blocos parcialmente sinterizados (usinagem de corte macio ou "*soft machining*") (DENRY; HOLLOWAY, 2010), os quais contraem cerca de 25% após sinterização total (DENRY; KELLY, 2008).

Acredita-se que o desgaste superficial das cerâmicas nos processos de usinagem possa atuar em duas direções principais e gerar dois efeitos antagônicos (KOSMAČ et al., 1999). Por um lado, tem sido relatado na literatura que a usinagem poderia produzir uma fina faixa de tensão compressiva sobre a superfície da cerâmica, dificultando a propagação de trincas e promovendo melhoria na resistência do material (AURÉLIO et al., 2015; GIORDANO; CIMA; POBER, 1995; MARSHALL et al., 1983). Discos usinados em vitrocerâmica leucítica e que não receberam nenhum tratamento (controle) apresentaram maiores valores de resistência flexural (187,7 MPa) se comparados aos discos submetidos à tratamento térmico de glazeamento recomendado pelo fabricante (153,7 MPa) (AURÉLIO et al., 2015). Os autores sugeriram que o tratamento térmico de glazeamento possa ter removido a camada de tensão compressiva deixada na superfície dos espécimes após a usinagem e associaram o decréscimo dos valores de resistência à possível perda do efeito protetor das tensões residuais compressivas.

Por outro lado, tem-se a indução de defeitos superficiais, os quais poderão atuar como áreas de concentração de tensões, reduzindo a resistência da cerâmica (FRAGA et al., 2015,

2017; KOSMAČ et al., 1999; QUINN, 2007). Sindel et al. (1998) estimaram em 40-60 µm a extensão dos danos da usinagem para uma cerâmica feldspática usinada pelo sistema CEREC1® (Sirona Dental Systems Gmbh, Alemanha), o qual permitia apenas a confecção de restaurações do tipo *inlay* por meio do desgaste de blocos cerâmicos com discos diamantados, sendo bem mais agressivo que os sistemas atuais que utilizam pontas diamantadas.

A análise fractográfica de barras de cerâmica feldspática e vítrea reforçada por partículas, submetidas a ensaio de flexão três pontos, mostraram que todas as barras falharam devido a defeito introduzido pela usinagem. O tamanho médio da falha que deu origem à fratura foi estimado em 9-15 µm para a cerâmica vítrea reforçada e 15-30 µm para a cerâmica feldspática (KELLY et al., 1991).

Em ensaio de flexão uniaxial, barras de zircônia parcialmente estabilizada por óxido de ítrio (Y-TZP) obtidas por usinagem pelo sistema Cercon (Degudent GmbH, Alemanha) apresentaram resistência característica de 820,65 MPa, valor inferior ao grupo que recebeu apenas polimento (1244,17 MPa). O valor médio de Ra após usinagem foi de 1,91µm, enquanto para os corpos de prova polidos foi de 0,04 µm (WANG; ABOUSHELIB; FEILZER, 2008).

Scherrer et al. (2011) estudaram o comportamento em fadiga de barras usinadas de Y-TZP após serem jateadas com partículas de óxido de alumínio revestidas por sílica de 30µm. Os autores constataram por meio de microscopia eletrônica de varredura (MEV) que as barras falharam a partir de defeitos intrínsecos e de danos promovidos pela usinagem.

Fraga et al. (2015) mostraram que a usinagem de corte duro reduziu em cerca de 27% a resistência flexural biaxial de discos usinados de cerâmica vítrea reforçada por leucita (128,20 MPa; IC 95%: 122,2-134,4 MPa), quando comparados a discos usinados e polidos (177,2 MPa; IC 95%: 166-189 MPa). Imagens em microscopia eletrônica de varredura (MEV) da secção transversal dos discos indicaram que a usinagem foi capaz de introduzir defeitos na superfície da cerâmica, os quais foram removidos com a realização do polimento. A rugosidade média do grupo usinado (Ra) foi de 1,37 ± 0,18 µm, enquanto do grupo usinado e polido foi de 0,04 ± 0,01 µm. Devido à usinagem, espécimes de Y-TZP, dissilicato de lítio e de leucita tiveram sua resistência flexural à fadiga reduzida respectivamente em 40% (Usinagem = 536.48 MPa vs. Usinagem + polimento = 894.50 MPa), 33% (Usinagem = 187.71MPa *vs.* Usinagem + polimento = 278.93MPa) e 29% (Usinagem = 72.61MPa *vs.* Usinagem + polimento = 102.55MPa) (FRAGA et al., 2017). Diante disso, acredita-se que a intensidade dos efeitos relativos ao processo de usinagem por instrumentos diamantados de

corte parece estar intimamente ligado ao grau de sinterização do material cerâmico a ser fresado (DENRY, 2013; FRAGA et al., 2017).

2.3 TRATAMENTO TÉRMICO EM MATERIAIS CERÂMICOS

2.3.1 Efeito do tratamento térmico sobre as propriedades mecânicas das cerâmicas e variáveis relacionadas

Regimes térmicos envolvendo procedimentos laboratoriais rotineiros, como o glazeamento, até protocolos mais complexos como os de recozimento (*annealing*), tem sido investigados em relação aos possíveis efeitos sobre a resistência mecânica de materiais cerâmicos (ASAI et al., 2010; BRACKETT et al., 1989; DENRY; HOLLOWAY; TARR, 1999; DONG et al., 1992; FAIRHURST et al., 1992; FISCHER et al., 2005; GIORDANO; CIMA; POBER, 1995; GRIGGS; THOMPSON; ANUSAVICE, 1996); inclusive em espécimes usinados (AURÉLIO et al., 2015; FRAGA et al., 2015; MORES et al., 2017).

Uma série de variáveis como a presença e distribuição dos defeitos superficiais e sub-superficiais (internos), o tipo e a magnitude das tensões transitórias e residuais e a rugosidade superficial parecem influenciar o comportamento mecânico da cerâmica (ALBAKRY; GUAZZATO; SWAIN, 2004). Dessa forma, pressupõem-se que maior será o efeito do tratamento térmico sobre o comportamento mecânico dos materiais cerâmicos, quanto maior for o número de variáveis concomitantemente influenciadas por ele, desde que os efeitos produzidos não sejam antagônicos entre si.

São controversas as informações encontradas na literatura no que tange aos possíveis efeitos de diferentes tratamentos térmicos sobre as propriedades mecânicas de materiais cerâmicos. Talvez isso se deva ao fato de não existirem protocolos de queima bem definidos, e pela grande variação na escolha da temperatura máxima, da duração do período à temperatura de manutenção (*dwell time*) e, das taxas de resfriamento (lenta ou rápida) entre os estudos, promovendo consequentemente resultados diversos e, por certas vezes, inconclusivos.

Alguns estudos defenderam a queima de glazeamento por esta aumentar a resistência (BRACKETT et al., 1989; CHEN et al., 1999; DE JAGER; FEILZER; DAVIDSON, 2000; GIORDANO; CIMA; POBER, 1995) e reduzir a rugosidade superficial (CHEN et al., 1999; DALKIZ; SIPAHI; BEYDEMIR, 2009; DE JAGER; FEILZER; DAVIDSON, 2000) da cerâmica. Segundo eles, o efeito de reforço do glazeamento estaria relacionado à diminuição

da profundidade e ao arredondamento da ponta das fissuras, o que funcionaria como mecanismo de tenacificação, ao dificultar a propagação das trincas responsáveis pela falha do material (HIRAO; TOMOZAWA, 1987). Contudo, cabe ressaltar que os achados descritos por Brackett et al. (1989), Chen et al. (1999) e Giordano, Cima e Pober (1995) foram associados ao protocolo de *overglaze*, o qual consiste na aplicação de uma fina película de porcelana de baixa fusão sobre a superfície externa da restauração, sendo o conjunto submetido a uma temperatura entre 20ºC e 60ºC abaixo da temperatura de queima da porcelana (YILMAZ; ÖZKAN, 2010). Assim, como sugerido por Chen e colaboradores (2010), os efeitos alcançados pelo tratamento estariam vinculados à camada de cerâmica adicional aplicada sobre superfície do material, e não à influência da queima propriamente dita.

O comportamento mecânico de restaurações usinadas (MORES et al., 2017) e de barras (MOHAMMADIBASSIR et al., 2017), ambas em dissilicato de lítio densamente sinterizado, que sofreram reglazeamento após o desgaste de suas superfícies glazeadas, foi estatisticamente semelhante ao de amostras somente glazeadas (controle) (MOHAMMADIBASSIR et al., 2017; MORES et al., 2017) e, glazeadas/desgastadas (MORES et al., 2017). Mores et al. (2017) ainda concluíram que não houve diferença nos valores de carga à fratura entre os grupos “glazeado” *vs.* “glazeado/desgastado”; pressupondo-se assim, que parece ter sido o glazeamento prévio *(overglaze)* de superfícies desgastas e, não o reglazeamento, que atuou de fato em benefício das propriedades mecânicas da cerâmica.

Contrariamente, estudos demonstram que o efeito do tratamento térmico, de maneira isolada, foi suficiente para influenciar positivamente as propriedades mecânicas dos materiais testados. Dong et al. (1992) reportaram um aumento significativo dos valores finais de resistência (160 e 180 MPa) de uma cerâmica injetável (IPS-Empress) submetida à tratamentos térmicos adicionais à injeção (queima da cerâmica de cobertura, queima de pigmentos e queima de glazeamento) em comparação aos que sofreram somente injeção (em torno de 120 MPa). Discos de porcelana (Duceram LFC Dentine DA1, Ducera, Rosbach v.d.H, Alemanha) apresentaram maior lisura de superfície e aumento na resistência à flexão após serem submetidos à tratamento térmico correspondente ao glazeamento (DE JAGER; FEILZER; DAVIDSON, 2000). Hung et al. (2008) simularam o desgaste clínico com broca diamantada em discos cerâmicos de dissilicato de lítio e realizaram, em alguns grupos após o desgaste, tratamentos térmicos correspondentes à queima da cerâmica de cobertura e ao glaze. Mesmo não ocorrendo mudanças significativas nos valores de rugosidade, os autores detectaram a ocorrência de cicatrização de trincas e formação de uma camada de vidro após o

tratamento térmico, comprovadas por fractografia. Além disso, os valores de resistência (inicialmente reduzidos devido ao processo de desgaste à valores em torno de 233,7 MPa) foram restaurados após a realização dos ciclos térmicos, atingindo valores ao redor de 276,1 MPa, os quais se equipararam aos valores do material previamente ao desgaste (283,2 MPa). Aumento significativo nos valores de resistência flexural de uma vitrocerâmica leucítica usinada foi alcançado por Aurélio et al. (2015) submetendo os espécimes somente ao ciclo térmico. Tal tratamento consistiu em prolongar para 15 minutos o tempo de manutenção da queima de glazeamento, seguido de resfriamento lento; enquanto a recomendação do fabricante indicava 1,5 minutos, seguido da abertura imediata do forno.

Outros trabalhos sugerem que o tratamento térmico não tem nenhum efeito sobre a resistência dos materiais cerâmicos (ADDISON et al., 2012; AHMAD et al., 2005; DENRY; HOLLOWAY; TARR, 1999; GRIGGS; THOMPSON; ANUSAVICE, 1996). No estudo de Ahmad et al. (2005), a resistência de barras de cerâmica feldspática reforçada por alumina, abrasionadas com disco diamantado de granulação 70 µm, não foi alterada após os espécimes terem permanecido à 940ºC por 2 minutos. Espécimes de cerâmica reforçada por leucita IPS Empress (Ivoclar AG-Schaan, Liechtenstein) submetidos à queimas adicionais à de injeção, que simulariam o glazeamento e a aplicação de pigmentos, não alcançaram valores de resistência estatisticamente diferentes se comparado às amostras que receberam apenas ciclo inicial de injeção (ÜÇTAŞLI; WILSON, 1996). Griggs, Thompson e Anusavice (1996) verificaram que o *self-glaze* não alterou a resistência de discos de porcelana contendo falhas induzidas por meio de indentação Vickers. No *self-glaze*, a restauração cerâmica é exposta, sem nenhum aditivo, a uma temperatura igual ou ligeiramente superior a sua temperatura de queima, pelo tempo de 1 a 2 minutos, a fim de promover o derretimento da sua camada mais superficial e atingir o brilho desejado (YILMAZ; ÖZKAN, 2010).

Protocolo térmico de *annealing* à 900ºC por 2 horas não foi efetivo na melhoria da resistência de espécimes polidos, obtidos da secção de blocos de cerâmica feldspática para sistemas CAD-CAM, mesmo tendo sido capaz de reduzir defeitos gerados via indentação Vickers nesse material (DENRY; HOLLOWAY; TARR, 1999). A rugosidade superficial e a resistência flerural de discos de cerâmica feldspática usinados não foram influenciadas após *annealing* realizado à 900ºC pelo tempo de 1 hora (ADDISON et al., 2012). O tratamento térmico conhecido como *annealing* ou recozimento é conduzido a uma temperatura constante, compreendida entre a temperatura de transição vítrea (T_g) e a temperatura de amolecimento (DENRY; HOLLOWAY; TARR, 1999) que tem sido aplicado, no caso das cerâmicas odontológicas, na tentativa de promover o alívio de tensões oriundas do processamento da

cerâmica (FISCHER et al., 2005), mas também, em alguns casos, visando promover cicatrização de defeitos (DENRY; HOLLOWAY; TARR, 1999).

Existem ainda, estudos que apontam para o detrimento das propriedades mecânicas dos materiais em função do tratamento térmico. Fairhurst et al. (1992) verificaram redução na resistência de discos de porcelana polidos, quando submetidos ao *self-glaze*. Dados recentes indicam que a queima de glazeamento tem efeito deletério sobre materiais vitrocerâmicos usinados. Ao que parece, o tratamento térmico teria removido as tensões compressivas deixadas pela usinagem, as quais seriam de caráter protetivo segundo alguns autores (GARVIE; HANNINK; PASCOE, 1975; MARSHALL et al., 1983) e explicariam os maiores valores de resistência apresentado por espécimes somente usinados se comparados à espécimes usinados e submetidos posteriormente ao ciclo de glazeamento (AURÉLIO et al., 2015; FRAGA et al., 2015). Por outro lado, o próprio glazeamento de porcelanas, mesmo quando na configuração de *self-glaze*, tem sido associado com a formação de uma camada de tensão compressiva protetora na superfície do material (FAIRHURST et al., 1992; GREEN; TANDON; SGLAVO, 1999; GRIGGS; THOMPSON; ANUSAVICE, 1996).

Independentemente do tipo de tensão, compressiva ou trativa, estudos demonstram que queimas envolvendo gradientes térmicos significativos devem ser evitadas (AL-AMLEH et al., 2014; THOLEY; SWAIN; THIEL, 2011), pois produzem tensões transitórias e residuais na superfície da cerâmica que induzem à propagação de trincas (BENETTI et al., 2014).

2.3.2 Efeito do tratamento térmico sobre a estabilidade das propriedades ópticas e da microestrutura cristalina das cerâmicas

Alguns trabalhos têm reportado a instabilidade de alguns componentes cerâmicos, na sua maioria óxidos metálicos e pigmentos (OZTURK et al., 2008; PIRES-DE-SOUZA et al., 2009), e da microestrutura cristalina dos materiais (YILMAZ; GONULDAS; OZTURK, 2014) frente ao tratamento térmico. As alterações da microestrutura, mesmo quando sutis, podem determinar novas propriedades mecânicas, químicas e físicas para um dado material, incluindo novas características ópticas (CALLISTER; RETHWISCH, 2012).

Yilmaz, Gonuldas e Ozturk (2014) avaliaram as alterações de cor de cerâmicas dentárias em função do tipo de material – IPS Classic metal-ceramic, Empress Esthetic e Empress 2; e dos métodos de glazeamento – glaze com aplicação de pasta; e glaze natural, caracterizado apenas pela queima e polimento; durante múltiplas queimas. Os autores constataram também

que a exposição à temperatura afetou negativamente a superfície da porcelana, com a deterioração da cor, destacando tons de amarelo e vermelho desde a primeira queima e ao longo das queimas subsequentes. As cerâmicas com maior teor cristalino foram as mais afetadas e, os processos de recristalização e desvitrificação de suas microestruturas frente ao tratamento térmico prolongado ou à altas temperaturas seriam, de acordo com os autores, os responsáveis pelas alterações de cor mais pronunciadas.

Queimas sequenciais produziram redução significativa nos valores da coordenada L* (luminosidade) do sistema CIEL*a*b* (*Comission Internacional l'Eclairage*) de espécimes à base de dissilicato de lítio (IPS e.max press) e foram atribuídas à possível instabilidade dos óxidos metálicos e pigmentos à ação da temperatura (OZTURK et al., 2008). Pires-de-Souza et al. (2009) também associaram as diferenças de cor encontradas para espécimes de vitrocerâmica IPS d.SIGN, submetidos à até quatro ciclos de queima à 900°C, à presença de óxidos metálicos na composição cerâmica, os quais seriam facilmente quebrados sob altas temperaturas. Uludag e colaboradores (2007) atribuíram o aumento das axiais a* e b* (amostras mais avermelhadas e amareladas, respectivamente) e a redução da luminosidade de espécimes de uma vitrocerâmica leucítica injetável ao comportamento instável dos aditivos presentes na composição das cerâmicas, responsáveis pela cor, após ciclos repetitivos simulando a queima da cerâmica de cobertura.

O tratamento térmico, dependendo da temperatura alcançada e do tempo de ação, também pode alterar a fração volumétrica das fases (MACKERT et al., 2003; MACKERT; EVANS, 1991, 1992; MACKERT; RUSSEL, 1996), modificar (BARREIRO; VICENTE, 1993) ou degradar (FRAGA et al., 2015) seus constituintes.

Barreiro e Vicente (1993), estudaram a cinética das transformações de fase da leucita em uma típica porcelana para a restaurações metalo-cerâmicas. Os autores lançaram mão de protocolos a partir de 800ºC, passando por 850ºC, 900ºC, 950ºC, a até 1000ºC, por períodos de tempo que variaram, para cada um dos ciclos térmicos, entre 10 até 1460 minutos (24 horas). Alguns dos tratamentos propostos, foram capazes de promover alterações das fases constituintes, como por exemplo, protocolo à 800ºC a partir de 60 minutos, o qual induziu a precipitação de sanidina (cristalina); sendo tal condição perpetuada durante os demais tratamentos à temperaturas mais elevadas.

No estudo de Fraga et al. (2015) discos usinados de uma vitrocerâmica reforçada por leucita, submetidos ao tratamento de glaze preconizado pelo fabricante (790ºC por 1,5 minutos), apresentaram distorção em um dos picos de leucita (difração de raios-X) indicando

presença de material amorfo e resistência flexural reduzida significativamente em relação à discos somente usinados.

Mackert e Russell (1996) simularam queimas de pigmentos e de glaze após a injeção em espécimes de vitrocerâmica reforçada por leucita (IPS Empress). Os resultados revelaram que o conteúdo de leucita não foi alterado após o processo de injeção (não houve cristalização adicional de leucita), entretanto, o mesmo aumentou significativamente após a primeira queima de pigmentos, e tal situação se perpetuou em queimas adicionais e nas queimas de glaze. Diminuições no conteúdo de leucita também foram medidos em procedimentos envolvendo queimas múltiplas de porcelanas dentárias (MACKERT; EVANS, 1991, 1992). Mackert e colaboradores (2003) investigaram o efeito de múltiplas queimas e de tratamento térmico que simulou a solda de infraestruturas metálicas recobertas por porcelana. Como resultado, a queima de solda produziu um aumento significativo na fração de leucita, enquanto o grupo de múltiplas queimas diminuiu significativamente o volume de leucita, quando comparadas ao grupo controle (sem tratamento térmico).

A microestrutura cristalina de cerâmicas à base de dissilicato de lítio, assim como no estudo de Kang, Chang e Son (2013), tem sido apenas descrita após a queima de cristalização dos materiais. Da mesma maneira, estudos acerca da microestrutura de compósitos à base de silicato de lítio reforçado por zircônia se restringem até o momento, a tão somente caracterizá-la (BELLI et al., 2016; KRÜGER et al., 2013), sem o efeito de qualquer variável associada. Deveras, os trabalhos que se propuseram a investigar as alterações da microestrutura de cerâmicas em função do tratamento térmico parecem ter sido direcionados em sua maioria às porcelanas dentárias.

2.4 FADIGA EM MATERIAIS CERÂMICOS

O termo fadiga é comumente usado para designar a falha de um material sujeito à deformações ao longo de um período de tempo (BARAN; BOBERICK; MCCOOL, 2001) que ocorrem sob tensões com intensidade abaixo da resistência nominal característica do material (WISKOTT; NICHOLLS; BELSER,1995). Em função de sua natureza frágil, a falha por fadiga das cerâmicas ocorre de maneira súbita e sem ser precedida de qualquer deformação plástica visível.

Vários testes laboratoriais permitem a avaliação do comportamento à fadiga de materiais cerâmicos, sendo que a seleção do tipo de ensaio, em função principalmente da aplicação da tensão, recai na informação que se deseja obter.

Cargas estáticas – nas quais um valor de tensão fixo é aplicado de maneira constante até a fratura (GONZAGA et al., 2011b) –, ou dinâmicas – onde a tensão é aplicada ininterruptamente durante o ensaio, sob uma taxa constante (GONZAGA et al., 2011b; THOMPSON, 2004) –, costumam ser vistas em metodologias que visam informações acerca do comportamento de crescimento de trincas dos materiais cerâmicos (GONZAGA et al., 2011b; THOMPSON, 2004). O crescimento subcrítico de trincas ou "*slow crack growth*" (SCG) (ZHU et al., 2003) ocorre na presença de umidade pela propagação das trincas em níveis de tensão mais baixos do que o nível crítico (abaixo do fator de intensidade de tensão crítica "K_{Ic}"), culminando na diminuição da resistência do material em função do tempo (GONZAGA et al., 2011a; TASKONAK et al., 2008) e, obviamente, na redução da sobrevida das restaurações (ZHU et al., 2003).

Cargas mecânicas cíclicas – nas quais a magnitude das tensões aplicadas e das deformações sofridas é variável ao longo do tempo (BARAN; BOBERICK; MCCOOL, 2001) –, têm sido comumente aplicadas sob condições de umidade para simular os desafios enfrentados pelas restaurações cerâmicas em uso e o dano acumulado que ocorre clinicamente (AMARAL et al., 2016; MAY et al., 2015; PEREIRA et al., 2016; ZUCUNI et al., 2017). Assim, a fadiga cíclica foi selecionada para o presente trabalho, uma vez que se objetivou avaliar o efeito que as queimas estendida e convencional de glazeamento teriam sobre o comportamento mecânico de vitrocerâmicas usinadas frente ao dano acumulado (resistência flexural à fadiga). O ensaio mecânico seguiu o método da escada, "*staircase*" ou "*up and down approach*", desenvolvido em 1948 por Dixon e Mood que confere uma característica de "sobe e desce" (escada) à plotagem dos resultados, em função das inversões que ocorrem entre os eventos de falha e sobrevivência ao longo do teste.

De acordo com Collins (1993), no ensaio proposto pelo método da escada incialmente um número de ciclos é previamente fixado (p. ex., 500.000 ciclos) para a aplicação de um valor de tensão inicial máxima, próximo do valor estimado para a resistência à fadiga do material – que comumente partirá de valores 30 à 50% abaixo da tensão obtida nos ensaios monotônicos de referência, dependendo do material testado. A próxima amostra será então submetida à ciclagem com um incremento fixo de tensão (normalmente corresponderá de 5 à 10% da tensão inicial), o qual será somado ou subtraído do valor de tensão estipulado anteriormente, caso o corpo de prova testado sobreviva ou falhe, respectivamente. Portanto, cada teste é dependente dos resultados do teste anterior.

No cenário atual, diversos pesquisadores têm lançado mão do método da escada para avaliar o comportamento à fadiga de materiais cerâmicos. Zucuni et al. (2017) investigaram

os efeitos de diferentes técnicas de obtenção de espécimes pré-sinterizados de cerâmica Y-TZP no comportamento à fadiga do material, com o intuito de responder se técnicas laboratoriais simplificadas teriam o desempenho mecânico correspondente ao de amostras de Y-TZP produzidas por usinagem CAD-CAM. Amaral et al. (2016) reportou a resistência à fadiga da cerâmica Y-TZP submetida à diferentes tratamentos de superfície (n=20) para diferentes números de ciclos (10^2, 10^3, 10^4 e 10^5), utilizando uma frequência de 0,5 Hz. Dibner e Kelly (2016) investigaram a resistência à fadiga de sistemas cerâmicos em função da razão formada entre as espessuras das camadas correspondentes às cerâmicas de infraestrutura e de cobertura (500.000 ciclos, 20Hz, n≥40). Fraga et al. (2016) avaliaram a influência da frequência de aplicação de carga (n≥20) – 2 Hz (controle), 10 Hz, 20 Hz, e 40 Hz – na resistência à fadiga de discos de Y-TZP após 500.000 ciclos. O efeito do desgaste com broca diamantada e da degradação hidrotérmica em autoclave no comportamento à fadiga de discos de cerâmica Y-TZP foi mensurado após 20.000 ciclos, sob frequência de 6 Hz, n=20 (PEREIRA et al., 2016). May et al. (2015) averiguaram a influência da espessura do cimento resinoso oclusal sobre as cargas cíclicas para a fratura (500.000 ciclos, com frequência de 20 Hz, n=20) de coroas feldspáticas usinadas e cimentadas à troqueis análogos de dentina. Belli et al. (2014) compararam a resistência à fadiga de resinas compostas e cerâmicas indicadas para restaurações parciais posteriores em configuração de ensaio de flexão uniaxial quatro pontos (10^4 ciclos, 0,5 Hz, n=25). Yi e Kelly (2011) avaliaram a resistência à fadiga por 500.000 ciclos, sob frequência de 15 Hz, n=15, de corpos de prova quadrangulares de cerâmica feldspática para CAD-CAM, submetidos à diferentes tratamentos de superfície e cimentados à bases de um material análogo à dentina.

A utilização de frequências baixas, entre 1 Hz e 2 Hz, próximas à do ciclo mastigatório (PO et al., 2011) torna a coleta dos dados muito lenta. Diante disso, alguns estudos têm sido realizados para verificar o efeito do aumento da frequência na resistência e nos parâmetros de fadiga de diferentes materiais (FRAGA et al., 2016; JOSHI et al., 2014; KELLY et al., 2010; ROSENTRITT et al., 2006). Fraga et al. (2016) concluíram que a utilização de frequências de até 20 Hz parece ser uma boa alternativa para agilizar os testes de fadiga cíclica de cerâmicas policristalinas. Kelly et al. (2010) investigaram o efeito da frequência na carga para fratura em fadiga de discos de cerâmica aluminizada infiltrada por vidro cimentados à bases de material análogo a dentina. Um pequeno aumento (cerca de 5 %) na carga para fratura em fadiga foi reportado para frequência de 20 Hz (602.5 N) em comparação aos resultados obtidos com 2 Hz (569.6N). Assim, a utilização de frequências na casa dos 20 Hz, de modo a acelerar o processo de dano acumulado foi indicada pelos autores.

3 ARTIGO 1 – EXTENDED GLAZE FIRING ON CERAMICS FOR HARD MACHINING: CRACK HEALING, RESIDUAL STRESSES, OPTICAL AND MICROSTRUCTURAL ASPECTS

Este artigo científico encontra-se publicado no periódico *Dental Materials* (v. 33, n. 2, p. 226-40, 2017), *Elsevier*, ISSN: 0109-5641, Fator de impacto = 4.070; Qualis A1. As normas para publicação estão descritas no Anexo A.

Extended glaze firing on ceramics for hard machining: Crack healing, residual stresses, optical and microstructural aspects

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ABSTRACT

Objectives: To evaluate the effect of extended and conventional (manufacturer–recommended) glaze firings on crack healing, residual stresses, optical characteristics and crystalline structure of four ceramics for hard machining.

Methods: Rectangular specimens were obtained by sectioning densely sintered feldspathic (FEL), leucite- (LEU), lithium disilicate- (DIS), and zirconia-reinforced lithium silicate-based (ZLS) prefabricated ceramic blocks and divided into groups according to the applied glaze firing (n=5): conventional glaze/manufacturer–recommended (G), extended glaze (EG) and control/no heat treatment (C). Defects generated by indentation were analyzed by scanning electron microscopy before and after firing (n=1) to evaluate crack healing. Residual stresses were determined by the indentation technique. Color differences (ΔE) after firing were measured by CIEDE2000 formula, and translucency variations were quantified by contrast ratio. Stability of crystalline microstructure was analyzed by X-ray diffraction.

Results: Regardless of the material, EG had greater ability than G to heal defects, and produced compressive residual stresses, while G generated tensile stresses. Color differences produced by EG were: imperceptible for FEL and LEU ceramics; perceptible, but still clinically acceptable for DIS; clinically unacceptable for ZLS. G produced no perceptible color change. The DIS and ZLS ceramics became ≈1% more opaque after G, ≈4% and ≈15%, respectively, after EG. The crystalline phase of all the ceramics remained stable after G and EG.

Significance: Extended glaze firing could be an alternative to finish feldspathic, leucite-, and lithium disilicate-based ceramic restorations, since it provides greater crack healing than the conventional glaze firing. It develops tolerable residual stresses, and produces clinically acceptable color alterations, without altering the microstructure of these materials.

KEYWORDS: Glass ceramic; Heat treatment; CAD/CAM; Stress state; Opacity; Microstructure analysis

1. Introduction

Feldspathic, leucite-, lithium disilicate- and lately, zirconia-reinforced lithium silicate-based ceramics [1] are materials available for so-called "hard machining" [2], in which densely sintered blocks are milled into the desired restoration format. Recently, Belli et al. [3] presented promising life estimates for posterior single unit all-ceramic hard-machined restorations: the expected time for 10% of the restorations to fail was ≥10.9 years. However, ceramic fracture still appears as one of the main technical failures or complications in clinical studies [4–8], and the brittle nature of these materials remains a challenge for the machining process [9,10].

The fabrication of glass ceramic restorations frequently includes the application of stain oxides and glazing for finishing, which are important for mimicking tooth structure and appearance [11]. According to Denry [9], the inability of manufacturer–recommended finishing procedures, such as glaze firings, to reduce the hard-machining damage indicates that large flaws produced by grinding during the milling stage may become fracture origins. Furthermore, some results suggest a possible deleterious effect of glaze firing on machined glass ceramic materials [11,12]. Therefore, there is a need for studies evaluating the feasibility of alternative firings that will maximize the performance of densely sintered ceramics for CAD/CAM systems.

With this intent, Aurelio et al. [11] extended the glaze firing dwell time at 790ºC from the manufacturer-recommended 1.5 min [13] to 15 min, and followed this by slow cooling, rather than immediate furnace opening, for a hard-machined leucite glass ceramic. The extended glaze had significantly increased the ceramic flexural strength compared to the conventional glaze firing. The authors credited this result in part to the possibility that the extended firing had induced a decrease in flaw depth and sharpness, increasing the stress needed for crack initiation.

Healing of defects in ceramic materials, either by thermal diffusion [14,15] or by oxidation mechanisms [16,17], has been previously studied. Some studies show the ability of certain heat treatments to reduce the viscosity of the glass matrix, causing a decrease in the depth of fissures, crack–tip blunting [18,19], and even sealing of defects [20,21] via capillarity [14,22]. Healing seems to be favoured by materials containing silicon (Si), which when subjected to the proper heat treatment,

go through an oxidation reaction and generate viscous SiO_2-based products (crystalline or amorphous) which tend to seal the defects [23,24].

Both the magnitude and the profile of residual stresses developed after thermal treatments seem to be important aspects that impact on the ceramics' mechanical behaviour. Studies demonstrate that firings involving significant thermal gradients should be avoided [25,26], since they produce transient and residual stresses on the ceramic surface that induce the propagation of cracks [27]. On the other hand, formation of a protective compressive stress layer on the material surface has been detected after glazing firings [18,19].

Ideally, firings should not interfere with the desired effect of glaze (e.g., brightness, smoothness) or the optical characteristics of the restoration. Thermal cycles must also meet the requirements for a structural balance of the glassy and crystalline phases, as slight variations in the microstructure resulting from firing may produce new mechanical, chemical or physical properties for a specific material [28]. Some studies suggest that, depending on the firing regime adopted, metal oxides responsible for the color of the material may become unstable [29,30] and that heat treatment may lead to alterations in the material constituent phases [12,31,32]. Therefore, it is necessary that studies compile information regarding the effect of heat treatments on the optical properties and microstructure of ceramics.

Given this context, the present study aimed to evaluate the effect of conventional (manufacturer–proposed) and extended glaze firings on crack healing, residual stresses, optical properties and crystalline microstructure of four ceramics intended for hard machining, having different microstructures. The first hypothesis was that an extended glaze firing would have a better ability to heal defects and that it would result in a compressive residual stress state, as compared to a conventional glaze firing. The second hypothesis was that the thermal cycles would not alter either the optical properties (beyond the clinically unacceptable established threshold) or the crystalline phase of the ceramic materials investigated.

2. Materials and methods

Descriptions of the microstructure, composition, and indications of the four ceramics for hard machining used in this study are given in Table 1.

2.1. Specimen preparation

Rectangular specimens were obtained by sectioning densely sintered prefabricated ceramic blocks (Table 1), with a diamond disc at low-speed, under water-cooling, in a sectioning machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA). The upper and lower surfaces of the specimens were flattened with 400 and 600 grit silicon carbide papers. One face then received mirror polishing, using 1200 and 2000 grit silicon carbide papers followed by 0-2 µm diamond polishing paste (Christensen Roder, Porto Alegre, RS, Brazil), resulting in specimens of 1.5 ± 0.5 mm thickness at the centre (210 MAP micrometer, Starrett, USA). All specimens were cleaned in an ultrasonic bath (1440 D - Odontrobras, Ind E Com Equip Med Dental LTDA, Ribeirão Preto, Brazil) using isopropyl alcohol for 10 minutes.

2.2. Experimental groups: glaze firings applied to the ceramics

Specimens of each ceramic material were randomly allocated to groups having designated firing protocols (Table 2). The control group (C) did not receive any heat treatment; while the conventional glaze (G) and extended glaze (EG) groups were fired in a VITA VACUMAT 6000 MP furnace (VITA – Germany). The G firings strictly followed the manufacturers' recommendations. The furnace was opened at the end of the heating stage, providing abrupt cooling. The extended schedules (EG) were conducted with the same initial temperature, pre-heating time, and temperature increase rate as the respective G regimens, but were differentiated by holding the specimens for a dwell time of 15 min [11] and by slow cooling, leaving the furnace closed until the temperature dropped to 200°C.

Distinct methodological approaches were delineated according to the objectives of each analysis. For the crack healing and optical property analyses, the same specimen was evaluated at two different times – before and after the firing (G or EG). For the residual stress and crystalline structure analyses, different specimens from G, EG and C groups were evaluated at a single time following treatment.

Five specimens per group (n = 5) were subjected to the analyses of residual stresses and optical properties. One additional specimen per group (n = 1) was used for the crack healing investigation and for microstructural analysis.

2.3. Crack healing analysis

Before firing (i.e., the control condition), two extra specimens per material had a mirror-polished face indented in a Vickers microhardness tester (Shimadzu Micro Hardness tester, HMV-G21 model, Shimadzu, Kyoto, Japan) with a load of 19.6 N for 20 seconds, which produces an acceptable crack pattern [33], as shown in Fig. 1. The specimens were metallized, and one crack emanating from the indentation was examined per specimen in a field emission scanning electron microscope (SEM-FEG, Inspect F50, FEI, Hillsboro, Oregon, USA) with a magnification of 3000×. The two indented specimens per material type were assigned to the G or EG group (n = 1); the metallization was then removed using acetone followed by ultrasonic cleaning in isopropyl alcohol for 10 min (Model 1440 D, Odontrobras, Ind, E Com, Equip, Méd, Odonto, LTDA, Ribeirão Preto, Brazil) so the samples could be subjected to their respective glaze firings. After firing, each specimen had the same crack re-examined (SEM-FEG, 3000x) in order to qualitatively verify the effect of the heat treatment on healing [20].

2.4. Residual stress analysis

The calculation of residual stresses developed in the ceramic surface after firing (n = 5), was determined by the indentation technique [34]. For this, the mirror-polished faces of different specimens from G, EG and C groups were subjected to three Vickers indentations (19.6 N for 20 seconds, as described in section 2.3.).

The fracture toughness K_{Ic} of the samples (in MPa·m$^{0.5}$) was estimated from measurement of the radial cracks formed by indentation, using the following equation proposed by Anstis et al. [35]:

$$K_{Ic} = k \left(\frac{E}{H}\right)^{0.5} \times \frac{P}{c^{3/2}} \quad (1)$$

where E is the elastic modulus, P is the applied load (in N), H is the Vickers hardness, given by $H = 1.8544P/a^2$ (in GPa), where a is the average indentation half-diagonal length (in m); c is the average radial crack length measured from the centre of the indentation (in m), and k is a constant equal to 0.016. Values used for E from the manufacturers' specifications are: 62 GPa for IPS Empress CAD, 95 GPa for IPS e.max CAD, and 95 GPa for VITA SUPRINITY. For VITABLOCS Mark II, E =69 GPa as reported by Fischer et al. [36].

Then, the residual stress σ_r of the ceramic surface (in MPa) after each heat treatment (G or EG) was calculated by inserting the data calculated using Eq. (1) in the next equation [37]:

$$\sigma_r = \frac{\mathrm{K}_{\mathrm{I}c} - \mathrm{K}'_{\mathrm{I}c}}{2\sqrt{\frac{c}{\pi}}} \tag{2}$$

where $\mathrm{K}_{\mathrm{I}c}$ and $\mathrm{K}'_{\mathrm{I}c}$ (in MPa·m$^{0.5}$), respectively, are the fracture toughness values of the control (no heat treatment) and heat treated ceramic material groups, and *c* is the average radial crack length (measured from the indentation centre) in the heat treated group (in m).

The radial crack length *c*, indentation half-diagonal length *a*, Vickers hardness, fracture toughness, and residual stress of each specimen were calculated as the arithmetic mean of the values obtained from the three indentations in the group.

2.5. Optical properties analysis

The color parameters of specimens were measured in the CIE L*a*b* system (*Commission Internationale de l'Eclairage*: L* (lightness); a* (green-red coordinate); b* (blue-yellow coordinate) with a spectrophotometer (SP60 - EX-Rite, Grand Rapids, Michigan, USA) before (control condition) and after the G or EG glaze firing. The device was set in analysis mode, using the CIE standard illuminant D65 and a 10º-observer angle. A coupling medium (glycerol $C_3H_8O_3$; Vetec Fine Chemicals Ltd, - Rio de Janeiro, Brazil), with a refractive index of 1.47 was used to minimize background light scattering from the specimen [38].

2.5.1. Color evaluation

The specimens were positioned on a white background [29,39] (Byko charts Cat. No. PA-2822, BYK-Gardner, Columbia, USA), and three measurements of the L*, a* and b* coordinates were taken for each specimen, before and after firing (n = 5). The average value of these three measurements was subsequently inserted in the calculation of ΔE. The color alterations were measured by the color difference formula, using the CIEDE2000 (ΔE_{00}) method (an improvement from its predecessor, CIELAB) [40], according to the following equation:

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)\right]^{1/2} \tag{3}$$

in which $\Delta L'$, $\Delta C'$, and $\Delta H'$ are the differences in lightness, chroma, and hue for a sample before and after firing, and R_T is a rotation function that accounts for the interaction between chroma and hue differences in the blue region. The weighting functions S_L, S_H, and S_C adjust the total color difference for variation in the location of the color difference pair in L', a', b' coordinates and the parametric factors K_L, K_C and K_H are correction terms for experimental conditions. In the present study, these parametric factors in the CIEDE2000 color difference formula were set to 1 ($K_L = K_C = K_H = 1$) [41,42] for the so-called reference conditions [43], which include homogeneous samples and the CIE standard illuminant D65.

The 50:50% perceptibility and acceptability thresholds [41] used to describe the results in this study were 1.25 and 2.23 units, respectively, as determined for dental ceramics using the TSK Fuzzy Approximation by Ghinea et al. [42].

2.5.2. Translucency evaluation

The specimens were placed on a white background and measurements of the L^* coordinate were repeated three times for each specimen (n = 5), in the before and after firing states. The same operation was also carried out using a black background (Byko charts Cat, No. PA-2822, BYK-Gardner, Columbia, USA). The average value of the three measurements made with both backgrounds for each specimen was then inserted in the contrast ratio formula (CR) [38,44]:

$$CR = Y_b / Y_w \tag{4}$$

$$Y = [(L^* + 16)/116]^3 \ x\ 100 \tag{5}$$

where Y_b denotes the reflectance over the black background and Y_w denotes the reflectance over the white background. In these calculations, CR = 0 is considered the most translucent and CR = 1, the most opaque.

2.6. Crystalline structure analysis

After treatment, one specimen from each of the groups (G, EG, and C) was subjected to X-ray diffraction (D8 Advance XRD, Bruker AXS GmbH, Germany) to evaluate the effect of firing on crystalline phase stability. For leucite-reinforced ceramic specimens (LEU), the measurement parameters were: scan range 20º to 45º, step size 0.009º, and a time per step of 87.5 s; for feldspathic (FEL), lithium disilicate- (DIS), and zirconia-reinforced lithium silicate-based (ZLS)

ceramics specimens: scan range 10º to 90º, step size 0.009º, and a time per step of 35 s. The wavelength used was 1.5416˚A (CuK_{α}). Peaks were identified from the crystalline structure values available for powder diffraction patterns (International Centre for Diffraction Data/Joint Committee for Powder Diffraction Studies).

2.7. Statistical analysis

The data collected regarding the radial crack length *c* and the fracture toughness were checked for normality of the distribution (Shapiro-Wilk, $P > 0.05$) and homogeneity of variances (Levene test, $P > 0.05$). The data that met both criteria were analysed using one-way analysis of variance (ANOVA), followed by Tukey's HSD test. Otherwise, a Kruskal-Wallis test followed by Fischer's LSD multiple comparisons test was used.

After confirming the normality and homoscedasticity of the data (Shapiro-Wilk, $P > 0.05$; Levene test, $P > 0.05$), the L^*, a^* and b^* coordinates and contrast ratio (CR) values were analysed by the paired *t*-test (dependent samples), and the statistical comparisons were restricted to each firing protocol. The Student *t*-test (independent samples) was selected to assess the color difference data (ΔE_{00}) except for LEU-G and LEU-EG groups, which were analysed by the Mann-Whitney test because of their non-parametric distribution ($P \leq 0.05$).

The study sample size provided a statistical power of at least 0.8 (80%) for all the analyses. All statistical tests were performed with a predetermined significance level of $\alpha = 0.05$ (SPSS version 17, SPSS, Chicago, IL, USA).

3. Results

Qualitative comparison of before and after microscopic images (Fig.2A–D and Fig.3A–D) shows the effect on crack healing of conventional (G) and extended (EG) glaze firings. The reduction in the defect size, although perceptible in both G and EG heat treatments, seems to be more pronounced for the EG firing, irrespective of the tested material.

The residual stress values (MPa) developed on the ceramic surfaces due to the heat treatments as well as the parameters directly involved in their calculation, i.e., radial crack length *c* and fracture toughness (units $MPa \cdot m^{0.5}$), are presented for each material investigated in Table 3. Regardless of the ceramic material, all groups subjected to the EG firing cycle developed compressive stresses (negative

values) on the ceramic surface (Table 3), and presented significantly lower crack length values and increased fracture toughness in comparison to the G and C treatments. In contrast, the conventional glaze firing cycle (G) induced tensile surface stresses (positive values) with magnitudes similar to the EG values; to complete the comparison noted earlier, the G firing produced significantly reduced fracture toughness and larger crack lengths in comparison to both EG and C for all the ceramics.

The effect of conventional (G) and extended (EG) glaze firings on the optical color coordinates L*, a*, and b* and the contrast ratio CR is shown in Table 4. For both G and EG firings, the DIS ceramic experienced statistically significant changes in all parameters: (L*, a*, b*, and CR), while the ZLS group showed changes in three parameters: (L*, a*, and CR). The LEU ceramic showed statistically changes in L* and a* for EG firing and in a* for G, while FEL showed a significant change only in L* (EG) or a* (G). The contrast ratio CR statistically increased toward 1 (greater opacity) after both conventional and extended glaze firings for both DIS and ZSL ceramics. The DIS and ZLS ceramics became ≈1% more opaque after G, ≈4% and ≈15%, respectively, after EG.

The overall effect of the glaze firing cycle on color alterations is seen in Table 5, where the (before–after) ΔE_{00} values calculated with the CIEDE2000 formula (Eq. 3) are shown. The data demonstrate that extended glaze firing (EG) induced a more pronounced color change than the conventional cycle (G) for all the ceramics (Table 5). The four ceramics subjected to the G firing achieved lower ΔE_{00} color alteration values than the reference perceptibility threshold ($\Delta E_{00} = 1.25$). With the EG firing, a color alteration that was perceptible but clinically acceptable was produced in the DIS specimens ($\Delta E_{00} = 1.76$), and clinically unacceptable in ZLS ($\Delta E_{00} = 3.22$). In the materials with higher vitreous content, such as the FEL and LEU ceramics, the EG firing produced an imperceptible color change when judged against the reference value $\Delta E_{00} = 1.25$.

The X-ray diffraction analysis revealed no changes in the materials' crystalline phases after heat treatment (Fig. 4A–D). The feldspathic (FEL) and leucite (LEU) ceramics remained stable crystallographically with peaks indicating crystalline structures corresponding to the feldspar family: sanidine ($(K.Na)(Si_3Al)O_8$, monoclinic) and nepheline ($NaAlSiO_4$, hexagonal) as shown in Fig. 4(A), and leucite ($KAlSi_2O_6$, tetragonal) in Fig. 4(B). The lithium disilicate- (DIS) and zirconia-reinforced lithium silicate-based (ZLS) ceramics also showed phase stability during the heat treatments. Peaks of

lithium disilicate ($Li_2Si_2O_5$, monoclinic) were found after G and EG firings in both ceramics DIS and ZLS (Fig. 4(C) and 4(D)) associated with lithium metasilicate peaks (Li_2SiO_3, orthorhombic) for ZLS (Fig. 4(D). Lithium phosphate (Li_3PO_4, orthorhombic) and lithium-cerium oxide (Li_8CeO_6, rhombohedral) crystalline formations, resulting from the chemical combination of binary oxides (LiO_2, P_2O_5, CeO_2) in the precursor materials, were also detected in the diffraction pattern from ZLS (Fig. 4(D)). The ZLS crystalline peaks appear to have become more intense as the glaze firing time was extended (Fig. 4(D)).

4. Discussion

The inability of glaze firings to potentially reduce the hard machining damage [9], and the possible deleterious effect of this cycle on machined glass ceramic materials [11,12] suggest the need for studies evaluating the feasibility of employing alternative thermal regimens. The study by Aurélio et al. (2015) [11] demonstrated that extended glaze firing significantly increased flexural strength, reduced surface roughness, and did not change the microstructure of leucite glass ceramic specimens after machining. Aligned with this approach, the present study investigated the effect of conventional (manufacturer–recommended) and extended glaze firings on healing of artificially-introduced defects and on residual surface stresses, using four hard machining ceramics with different microstructures. The effect of these firings on their optical properties and crystalline structures was also evaluated.

One of the hypotheses tested was that extended glaze firing would induce additional healing of artificially-created defects on the ceramic surface in comparison to conventional firing. The microscopy images (Fig. 2 (A–D) and 3 (A–D)), showing the area of the defect before and after heat treatment, demonstrate that the defect healing seems to be more pronounced with extended glaze firing (EG) in all of the ceramics treated in comparison to conventional glaze firing (G). This result is consistent with the hypothesis presented, and it can be credited to the prolonged period (15 min) during which the ceramics are allowed to remain at the maximum temperature. Such conditions may favour a possible oxidation reaction of the silicon in the ceramic [23,24], generating viscous products based on SiO_2 (silica), which tend to seal the defects. The extended dwell time, during which the ceramic material is above its glass transition, may keep the glass matrix viscosity low over sufficient time for viscous flow to reduce the flaw depth and change crack tips from sharp to round [18,19]; it

may also fully heal some areas [20,21] via capillarity [14,22]. In contrast, conventional glaze firing (G), regardless of the material tested, may allow insufficient time at the maximum temperature (from 1 to 1.5 min) for defect healing to occur.

The microscopy images, indicating some improved crack healing with extended glazing regimens, provide support for the results from Aurélio et al. [11], who reported higher flexural strength (211.7 MPa) in specimens of leucite-based glass-ceramic after extended glaze firing, in comparison to the glaze firing recommended by the manufacturer (153.7 MPa). According to Greil [23], besides increasing the toughness of the material, the concept of crack healing is an emerging approach to significantly improve the performance and reliability of ceramic components. A strategy proposed by Fischer, Weiss and Telle [45] suggests that compressive residual stress fields can be developed around the healed areas and deflect crack propagation. Certainly, the apparently promising results achieved by EG cycles in healing artificially-created defects in ceramic surfaces deserves detailed attention.

Regarding the influence of firings on the development of residual stresses in the ceramic surface, the hypothesis investigated was that extended glaze firing (EG), in comparison to conventional firing (G) would result in a state of compressive residual stress, which some studies claim is protective [11,12,46]. In fact, for all the ceramics analysed in the present study, EG resulted in the formation of a compressive stress layer in the material (Table 3), confirming the tested hypothesis. Such stress conditions promote the reduced propagation of cracks that was observed after an EG cycle in all materials, indicated by the decreased crack length *c* and increased fracture toughness compared to the control specimens (C) (Table 3). In the G specimens, in contrast to the EG, the tensile residual surface stresses observed were associated with larger crack lengths *c* and a decrease in fracture toughness, indicating easier propagation of cracks. The fracture mechanical property “critical stress intensity” (fracture toughness K_{Ic}) is defined by the resistance of a material against a propagating crack, being a characteristic, i.e. constant value for a specific ceramic material that does not exhibit rising toughness curve behavior [47]. So, since the X-ray diffraction analysis revealed no changes in the materials’ crystalline phases after firing (Fig. 4A–D), it is expected that observed differences in apparent fracture toughness between the groups (Table 3) were entirely caused by residual stress.

Studies suggest that treatments inducing significant thermal gradients should be avoided [25,26] since they produce transient and residual stresses on the ceramic, which can induce crack propagation [27]. However, the magnitude of the residual stress values found in this study, after G and EG firings, from -12.19 MPa (compression) up to 14.46 MPa (tensile), can be considered tolerable and not deleterious to performance when compared to the data of Benetti et al. [27]. Those authors observed that defects in the area surrounding failed crowns showed residual stresses from 40 MPa to 50 MPa; simulated stresses in the ceramic surface around 15 MPa using finite element analysis could not be considered high enough to promote sub-surface cracking.

As the present study, many others [26,37,48–51] have selected the Vickers indentation fracture toughness test [34] using the equation of Anstis et al. [35] for determining residual surface stress by differences in the materials' apparent fracture toughness. Mainly because of the simplicity and convenience of this method, causing minimal surface damage, without the need for special specimens or preparations [26,52]. Although the indentation technique is not consider an accurate or a reliable method for determining K_{Ic} [53,54], the data gathered in this study are precise, considering the low values of standard deviation of the groups. Also, since residual stress was calculated by the difference in fracture toughness estimates, any systematic lack of accuracy in fracture toughness was probably cancelled out. So, while the indentation technique may not be satisfactory for absolute ranking of different materials, it can give information about crack formation and comparative surface residual stress trends in a group of samples within a study [26].

The search for thermal cycles that maximize the performance of hard-machined ceramic restorations is justified only if they do not degrade the optical properties of the material and hence the desired aesthetic characteristics. In this study, we hypothesized that the firings would not affect the optical properties of the ceramics, regardless of the material microstructure. The hypothesis was partially confirmed, since ZLS subjected to the EG cycle had a color change (ΔE_{00} = 3.22; Table 5) higher than the acceptability threshold (ΔE_{00} = 2.23 [42]). For feldspathic (FEL) and leucite-based (LEU) ceramic, translucency was not altered, as no statistical difference was detected in the contrast ratio (CR) (Table 4), and color differences calculated by the CIEDE2000 method (Table 5) were considered imperceptible [42]. For the DIS and ZLS groups, on the other hand, the extended glaze

firing significantly increased the CR values of these ceramics (with less glass content), diminishing the translucency (CR values closer to 1) (Table 4). Despite the statistically significant increase in CR detected for the lithium disilicate-based ceramic (DIS) after EG firing (≈4%), the ceramic color alteration (ΔE_{00} = 1.76, Table 5) although perceptible (ΔE_{00} > 1.25), was still clinically acceptable (ΔE_{00} < 2.23). In spite of the promising crack healing results, EG firing is contraindicated for ZLS ceramics because the color changes (ΔE_{00} = 3.22) exceed clinical acceptability.

The color differences ΔE_{00} (Table 5) seen after thermal regimens express a composite of changes in individual color coordinates and in the larger number of parameters altered, particularly for the DIS and ZLS ceramics (Table 4). This finding can be attributed to the lower glassy content in the DIS and ZLS ceramic microstructures, which requires compensating additives (metal oxides, coloring ions) to control optical effects such as opalescence, color and opacity. These oxides tend to be unstable to the effect of elevated temperature [29,30], possibly clarifying why the extended glaze firing cycle (EG) produced significantly greater color changes for all ceramics (Table 5) than the much shorter G firing. On the other hand, the FEL and LEU ceramics do not require quantities of metal oxides and other additives, since the optical characteristics are inherent to their predominantly glassy formation [55] and are less sensitive to alterations in the optical properties related to firing.

Finally, possible microstructural changes during firing were investigated. We expected no modification of the ceramic materials' crystalline phase. The identified structures remained stable (Fig.4A–D), with no crystalline phases formed or degraded of existing phases, confirming the hypothesis investigated. The absence of zirconia crystalline phases in the ZLS-G and ZLS-EG groups (Fig. 4D) indicates that the ZrO_2 remains amorphous and aggregated to the glass matrix [56]. The fact that the heat treatments did not cause changes in the crystalline phase of the material shows that the cycles appear not to modify the microstructure established by the manufacturer. The relevance of such information is primarily to EG firing, which generally showed promise in maximizing the performance of hard-machined ceramics, without modifying the original ceramic structure. The exception was the ZLS ceramic, for which the results were aesthetically unacceptable. The more intense crystalline peaks [57,58] found for the ZLS ceramic after EG firing (Fig. 4D) could also be related to the material color instability due to possible microstructural changes other than phase

transformations. According to Yılmaz, Gonuldas, and Ozturk [59], continuous and/or high temperature firings in silicate-based materials could cause pyroclastic stream, recrystallization and devitrification. As a result, color changes could reach unacceptable limits.

Extended glaze firing could be an alternative to finish feldspathic, leucite-, and lithium disilicate-based ceramic restorations, since it: provides greater crack healing when compared to conventional glaze firing; develops tolerable residual stresses; produces color alterations within the clinical acceptability threshold; does not alter the material microstructure. However, despite the promising results reported by Aurélio et al. [11] on mechanical strength, further studies using hard-machined specimens associated to cyclic fatigue methodologies – that will simulate the realistic effects of automated grinding and the damage accumulation that occurs clinically – are also needed for the better understanding of the influence of these alternative thermal treatments. This is a possible future direction in the investigation of extended glaze firing in optimizing the performance of hard-machined restorations.

ACKNOWLEDGEMENTS

This study was partially funded by CAPES/CNPq.

The authors thank Professors Dr. Carlos Alexandre dos Santos and Dr. Felipe Dalla Vecchia, the Engineering Department Materials Laboratory (LAMAT) team at the Pontifical Catholic University of Rio Grande do Sul (PUCRS), and the Health Sciences Center at the Federal University of Santa Maria for supporting this research.

The authors confirm that there is no conflict of interest.

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FIGURES AND TABLES:

Figures

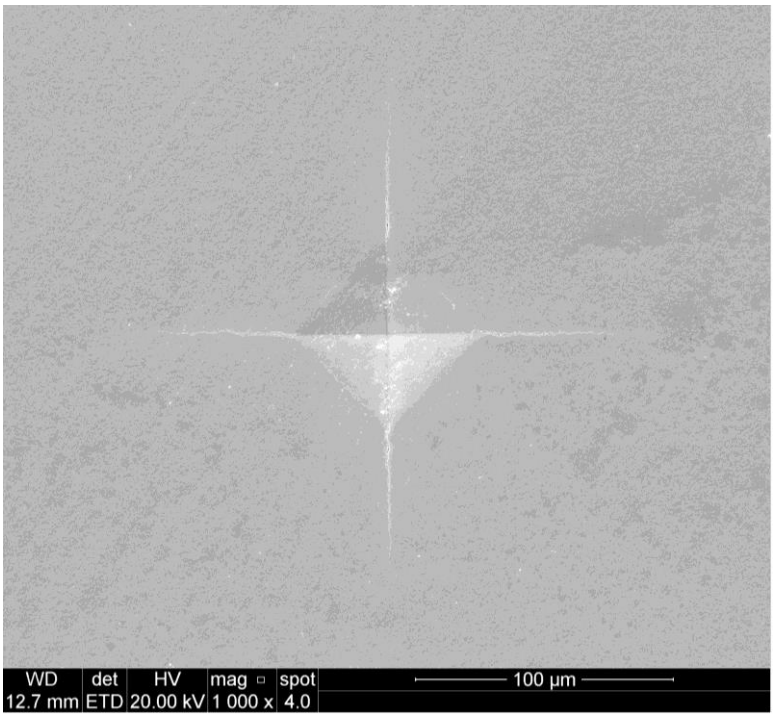


Fig. 1 – An acceptable crack pattern, using the criteria described by Nemli et al. [33]. Produced on a specimen of a lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) by indentation on its mirror-polished face (Vickers; 19.6 N for 20 seconds) before firing (control condition). One crack emanating from the indentation was chosen per specimen for defect healing analysis (n = 1).

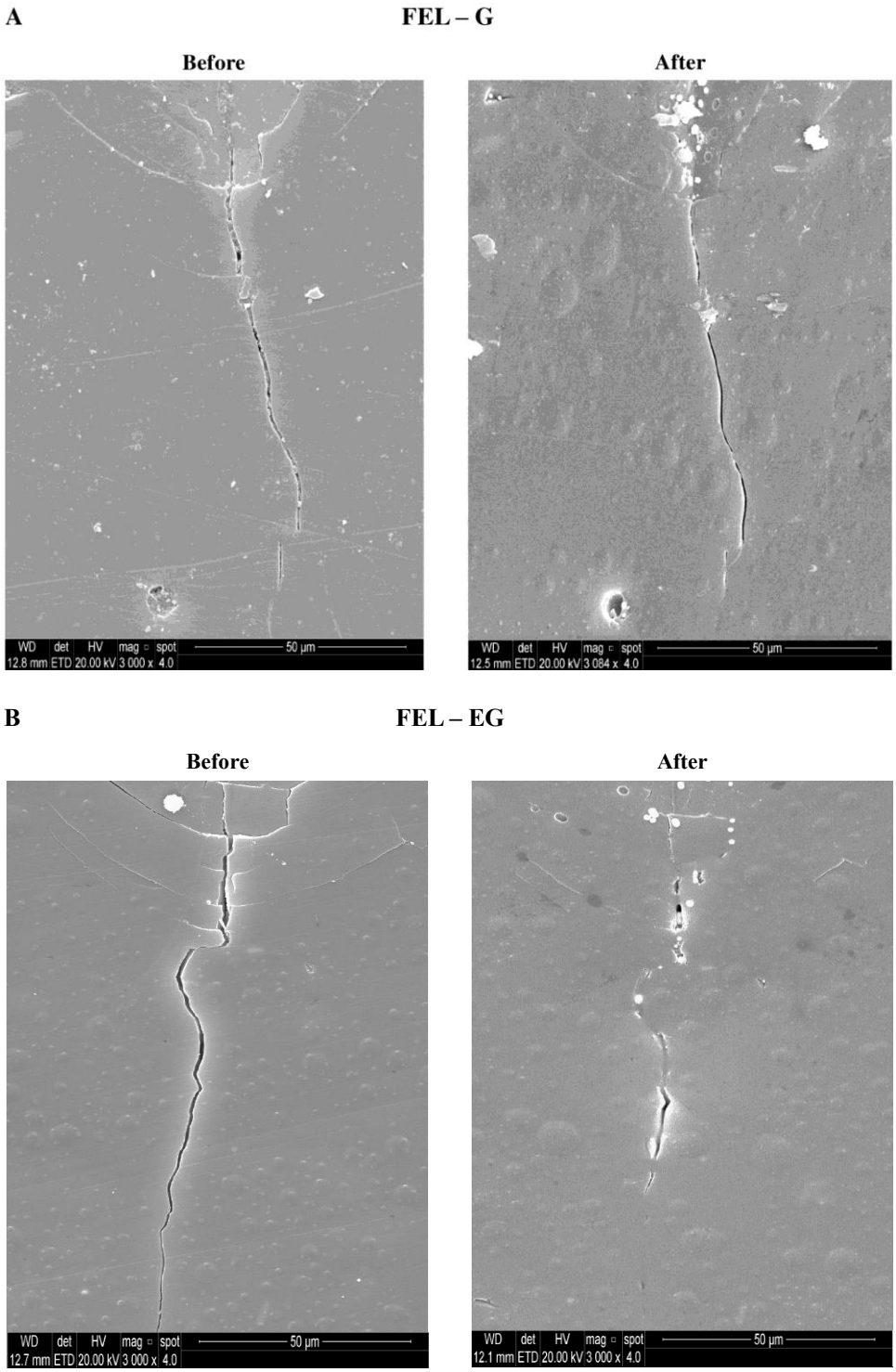
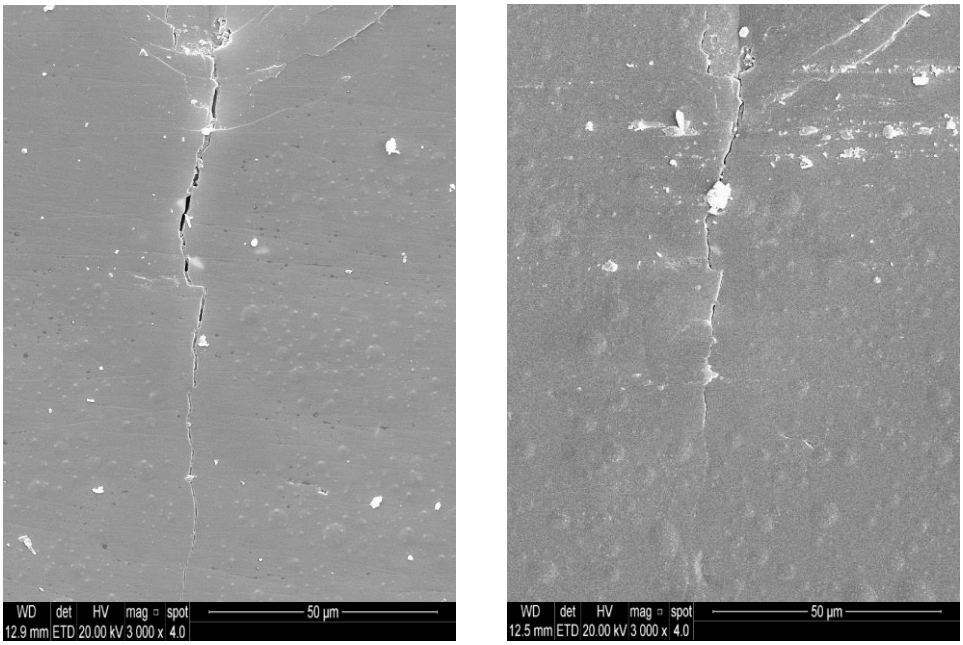


Fig. 2 – Crack healing: effect of conventional (G) and extended (EG) glaze firings on healing of an artificially-created defect. Microscopic images captured before (left) and after (right) the heat treatment. (A, B) Feldspathic (FEL) and (C, D) leucite-based (LEU) ceramics; G and EG firings are indicated. Gas bubbles inherent to the unconditioned glassy phase [60] are perceptible on the ceramic surface and were intensified by the heat treatment.

C **LEU – G**

Before **After**



D **LEU – EG**

Before **After**

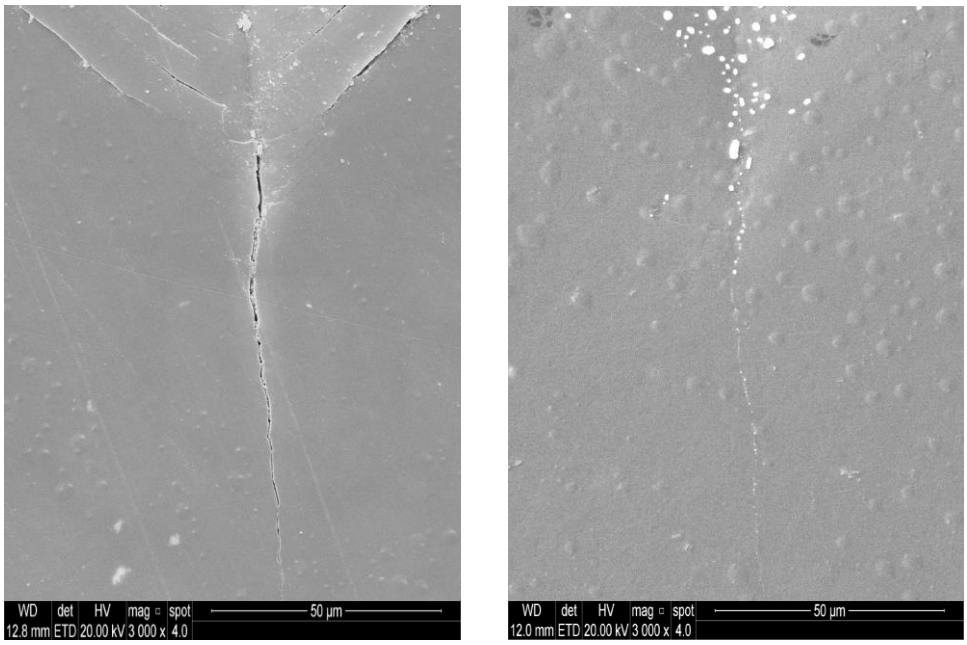


Fig. 2 – ***(Continued)***

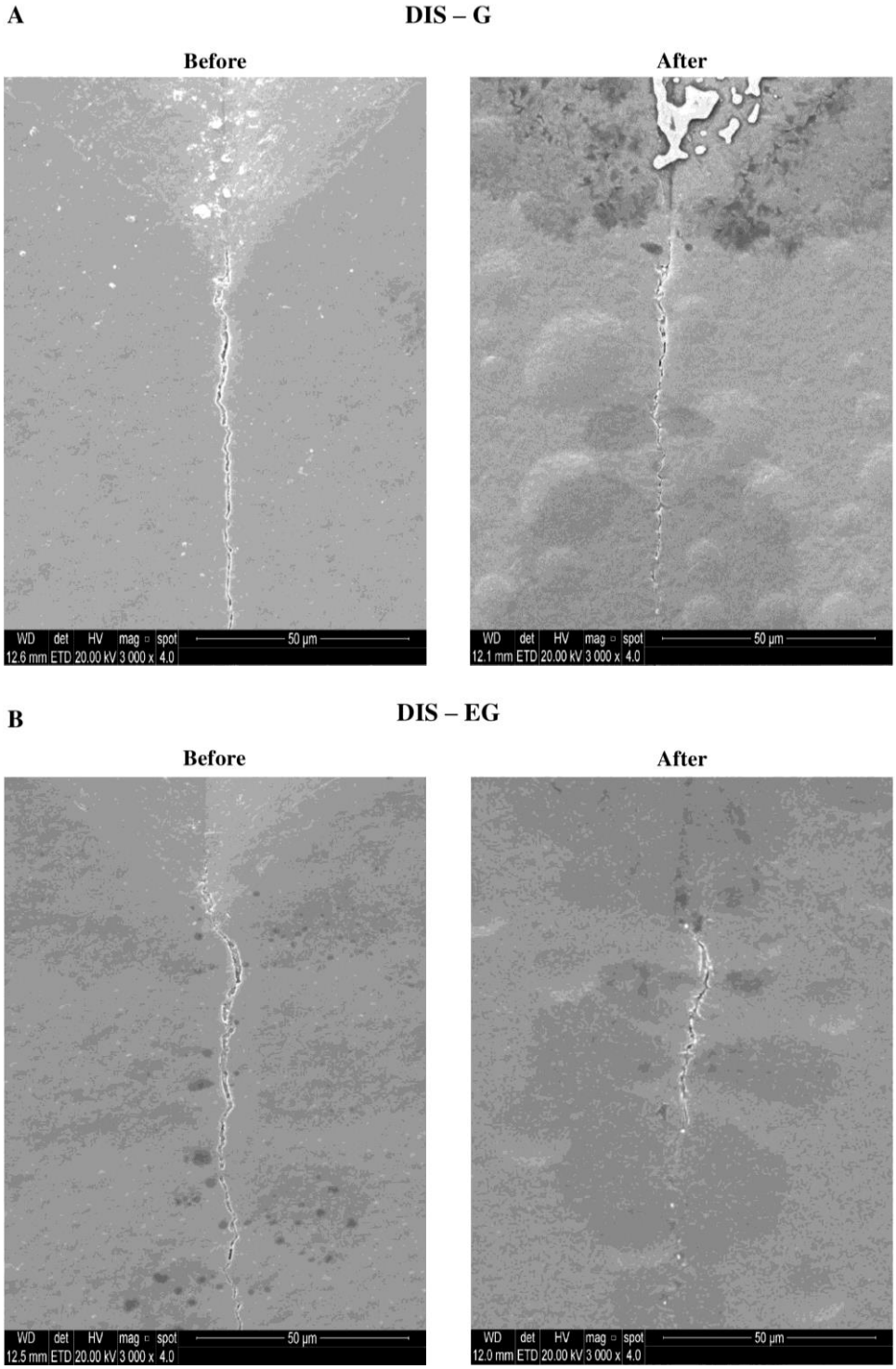
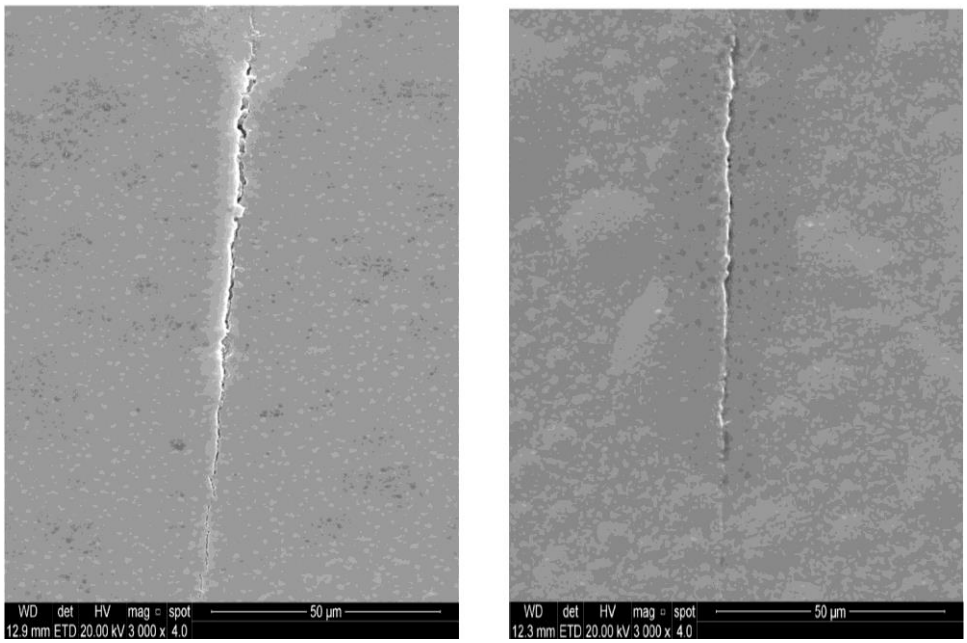


Fig. 3 – Crack healing: effect of (G) and (EG) glaze firings on an artificially created defect. Similar to Fig. 3, except: (A, B) Lithium disilicate- (DIS) and (C, D) zirconia-reinforced lithium silicate-based (ZLS) ceramics.

C **ZLS – G**

Before **After**



D **ZLS – EG**

Before **After**

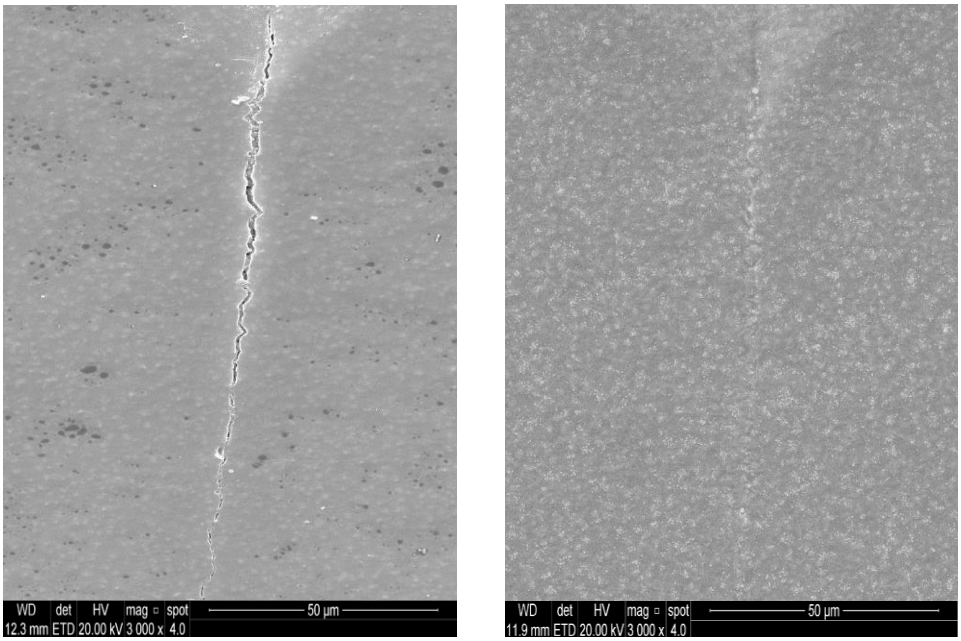


Fig. 3 – ***(Continued)***

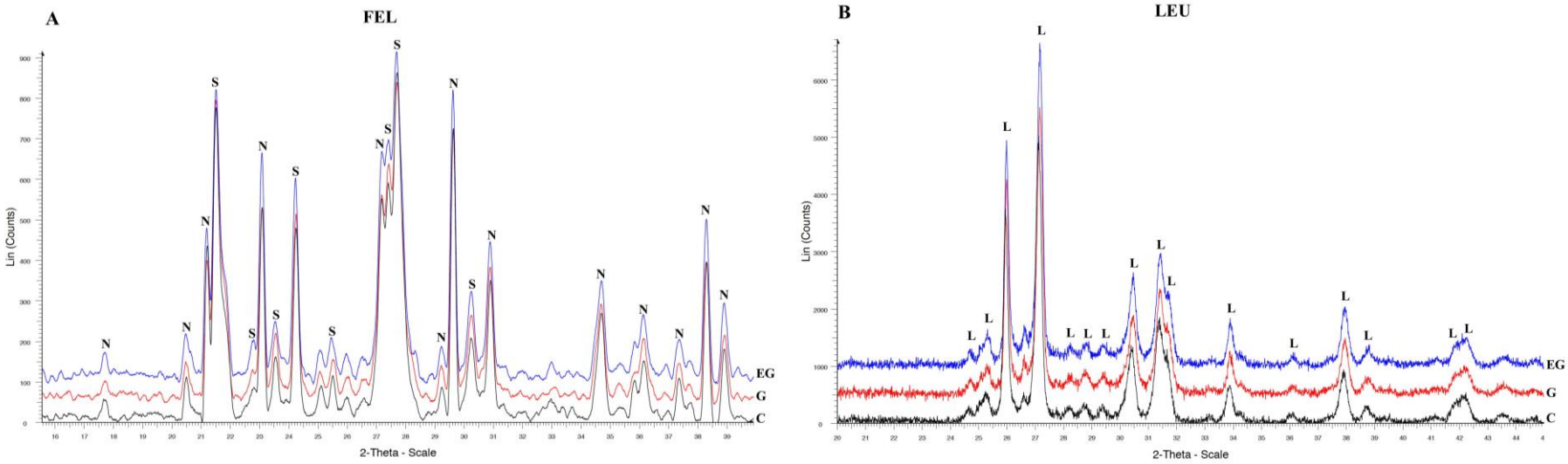


Fig. 4 – X-ray diffraction plots for the four ceramics, subjected to conventional (G) and extended glaze (EG) firings, or unfired control (C); traces for C, G and EG are offset vertically in each panel. (A) Feldspathic ceramic (FEL), (B) leucite ceramic (LEU), (C) lithium disilicate-based ceramic (DIS), and (D) zirconia-reinforced lithium silicate ceramic (ZLS). Crystalline peaks identified (*International Center Diffraction Data/Joint Committee for Powder Diffraction Studies*) are: N = nepheline (00-035-0424), S = sanidine (00-019-1227), L = leucite (01-071-1147), LD = lithium disilicate (01-072-0102), LM = lithium metasilicate (00-030-0766), LP = lithium phosphate (00-015-0760), and LCO = lithium cerium oxide (01-078-0343).

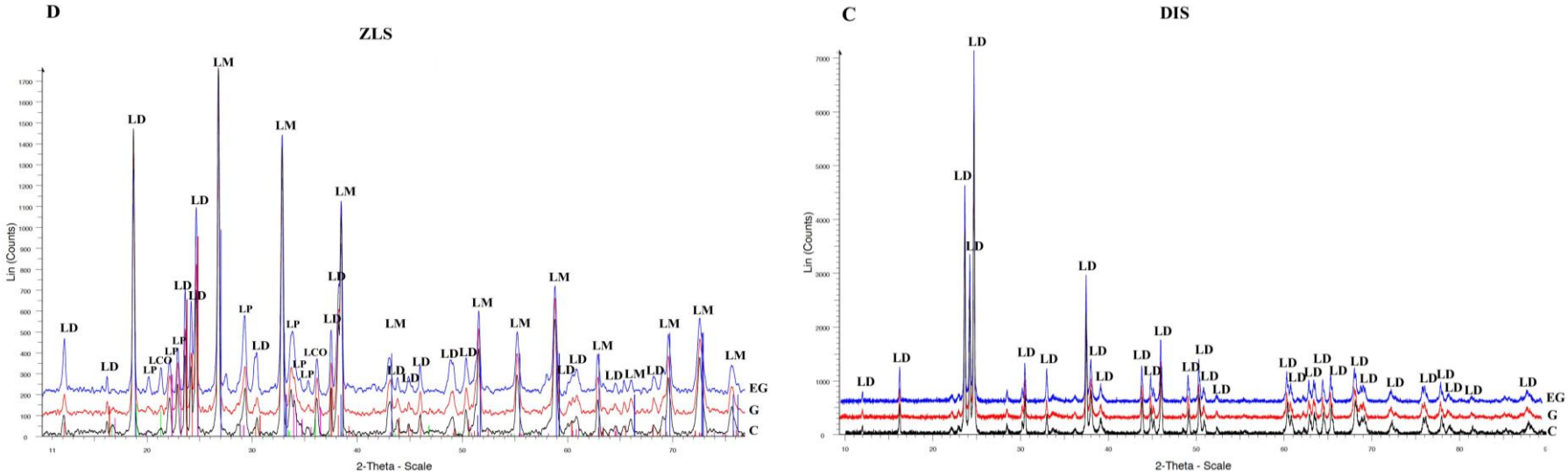


Fig. 4 – ***(Continued)***

Tables

Table 1 – Ceramics for hard machining used in the study.

Ceramic	Code	Color	Lot	Manufacturer	Microstructure	Composition[a]	Indications[a,b]
VITABLOCS Mark II	FEL	2M2C	45080	Vita Zahnfabrik, Bad Säckingen, Germany	Feldspathic ceramic. Feldspathic crystals (sanidine - $KAlSi_3O_8$; nefeline - $NaAlSiO_4$; albite - $NaAlSi_3O_8$) < 20%vol surrounded by a glassy matrix	Al_2O_3; SiO_2; K_2O e Na_2O; CaO; TiO_2; other oxides	Inlays, onlays, veneers, endodontic crowns (molars only), anterior and posterior single crowns and CAD/CAM veneering technique
IPS Empress CAD	LEU	A1-HT	S54980	Ivoclar-Vivadent, Schaan, Liechtenstein	Leucite glass ceramic. Leucite crystals ($K_2O.Al_2O_3.4SiO_2$) ≈35-45%vol surrounded by a glassy matrix	Al_2O_3; SiO_2; K_2O; Na_2O; other oxides; pigments	Inlays, onlays, veneers, partial crowns, anterior and posterior single crowns
IPS e.max CAD	DIS	A2	T33444	Ivoclar-Vivadent, Schaan, Liechtenstein	Lithium disilicate glass ceramic. When fully crystallized: lithium disilicate crystals ($Li_2Si_2O_5$) ≈70%vol surrounded by a glassy matrix	SiO_2 ; Li_2O; K_2O; P_2O_5; ZrO_2; ZnO; Al_2O_3; MgO; other oxides	Inlays, onlays, veneers, anterior and posterior single crowns, and three-unit bridges (up to the second premolar as the terminal abutment). Single abutments and single abutment crowns for anterior and posterior teeth. CAD/CAM veneering technique
VITA SUPRINITY	SLZ	A2	51250	Vita Zahnfabrik, Bad Säckingen, Germany	Zirconia-reinforced lithium silicate glass ceramic. When fully crystallized: lithium disilicate ($Li_2Si_2O_5$) and lithium metasilicate crystals (Li_2SiO_3) [1] surrounded by a glassy matrix containing zirconium oxide in solution (ZrO_2) ≈10%vol [56]	SiO_2 ; Li_2O; K_2O; P_2O_5; ZrO_2; Al_2O_3; CeO_2; pigments	Inlays, onlays, veneers, partial crowns, anterior and posterior single crowns. Anterior and posterior crowns on implant abutments

[a] The chemical compositions are described according to the manufacturers information.

[b] The indications are not associated to the specific ceramic color selected for the study.

Table 2 – Description of the experimental groups according to the treatments applied to the ceramic materials.

	Groups (n = 5)											
	FEL-G	**LEU-G**	**DIS-G**	**ZLS-G**	**FEL-EG**	**LEU-EG**	**DIS-EG**	**ZLS-EG**	**FEL-C**	**LEU-C**	**DIS-C**	**ZLS-C**
Ceramic code	FEL	LEU	DIS	ZLS	FEL	LEU	DIS	ZLS	FEL	LEU	DIS	ZLS
Glaze firing	Conventional (manufacuterer – recommended) glaze firing (G)				Extended glaze firing (EG)				No heat treatment (Control)			
Initial temperature (°C)	500	403	403	400	500	403	403	400	-			
Temperature increase rate (°C/min)	80	100	60	80	80	100	60	80	-			
Final temperature (°C)	950	790	770	800	950	790	770	800	-			
Dwell time at the final temperature (min:s)	1:00	1:30	1:30	1:00	15:00	15:00	15:00	15:00	-			
Vacuum	No	From 450°C to 789°C	From 450°C to 769°C	No	No	From 450°C to 789°C	From 450°C to 769°C	No	-			
Cooling rate	Fast. Immediate furnace opening after the dwell time				Slow. The furnace remained closed until the material reached 200°C				-			

Table 3 – Mean values and (standard deviation) for the radial crack length *c* (μm) and fracture toughness (MPa·m$^{0.5}$), determined by Vickers indentation. The residual stress (MPa) developed in the ceramic surface after thermal cycle, calculated according to Eq. (2), is described by mean (standard deviation) for each material and firing.

Groups (n = 5)		**Radial Crack Length c (μm)**	**Fracture Toughness (MPa·m$^{0.5}$)**	**Residual Stress (MPa)**
FEL	**-C**	122.85 (3.67)^{b}	0.72 (0.03)^{b}	-
	-G	141.99 (2.15)^{a}	0.59 (0.02)^{c}	9.41(1.25)
	-EG	113.67 (2.03)^{c}	0.83 (0.03)^{a}	-8.8 (2.50)
LEU	**-C**	92.55 (2.68)^{b}	1.10 (0.04)^{b}	-
	-G	102.64 (1.16)^{a}	0.93 (0.02)^{c}	14.46 (1.30)
	-EG	85.57 (1.01)^{c}	1.23 (0.01)^{a}	-12.19 (1.46)
DIS	**-C**	86.36 (1.22)^{b}	1.53 (0.03)^{b}	-
	-G	90.15 (0.58)^{a}	1.43 (0.01)^{c}	9.22 (1.14)
	-EG	82.29 (0.55)^{c}	1.63 (0.02)^{a}	-9.45 (1.72)
ZLS	**-C**	126.10 (3.44)^{b}	0.72 (0.03)^{b}	-
	-G	136.16 (2.10)^{a}	0.64 (0.01)^{c}	6.34 (0.86)
	-EG	115.96 (2.00)^{c}	0.80 (0.02)^{a}	-6.38 (1.56)

Different superscript letters denote significant differences ($P \leq 0.05$). Statistical comparisons are restricted to each ceramic material.

Table 4 - Mean and (standard deviation) of the L*, a*, and b* coordinates and contrast ratios before (control) and after the conventional (G) and extended glaze (EG) firings for each ceramic material.

Groups (n = 5)	Treatment Stage	L*	a*	b*	CR
FEL-G	Before	76.12 (0.07)[a]	1.55 (0.22)[a]	19.72 (0.15)[a]	0.69 (0.01)[a]
	After	76.29 (0.16)[a]	1.44 (0.18)[b]	19.59 (0.12)[a]	0.69 (0.01)[a]
LEU-G	Before	77.99 (0.42)[a]	-0.28 (0.07)[b]	13.54 (0.19)[a]	0.65 (0.01)[a]
	After	77.88 (0.32)[a]	-0.25 (0.06)[a]	13.42 (0.20)[a]	0.65 (0.01)[a]
DIS-G	Before	74.46 (0.49)[a]	1.20 (0.07)[b]	19.12 (0.18)[b]	0.75 (0.02)[b]
	After	73.61 (0.41)[b]	1.60 (0.08)[a]	19.59 (0.11)[a]	0.76 (0.01)[a]
ZLS-G	Before	69.93 (0.57)[b]	3.65 (0.08)[a]	21.05 (0.58)[a]	0.79 (0.02)[b]
	After	70.92 (0.66)[a]	3.26 (0.11)[b]	20.88 (0.44)[a]	0.80 (0.02)[a]
FEL-EG	Before	74.98 (0.21)[b]	1.62 (0.25)[a]	19.80 (0.16)[a]	0.70 (0.01)[a]
	After	75.35 (0.24)[a]	1.53 (0.19)[a]	19.88 (0.17)[a]	0.70 (0.01)[a]
LEU-EG	Before	77.54 (0.52)[b]	-0.29 (0.07)[a]	13.19 (0.24)[a]	0.66 (0.02)[a]
	After	78.17 (0.45)[a]	-0.37 (0.04)[b]	13.13 (0.22)[a]	0.66 (0.01)[a]
DIS-EG	Before	73.96 (0.23)[a]	1.25 (0.05)[b]	19.02 (0.13)[b]	0.76 (0.01)[b]
	After	72.48 (0.30)[b]	2.18 (0.03)[a]	20.96 (0.17)[a]	0.79 (0.01)[a]
ZLS-EG	Before	69.85 (0.85)[b]	3.54 (0.06)[b]	21.11 (0.46)[a]	0.78 (0.01)[b]
	After	73.85 (0.71)[a]	4.59 (0.06)[a]	21.05 (0.30)[a]	0.90 (0.01)[a]

Different superscript letters denote significant before-after differences ($P \leq 0.05$). Statistical comparisons for L* a* and b* coordinates and for the contrast ratio are limited to each firing protocol and presented a dependency relationship. Thus the same specimen is evaluated at two different instants and data collected prior to treatment serves as the control group.

Table 5 – Color alterations induced by conventional (G) and extended (EG) glaze firings for each ceramic material, determined by ΔE_{00} (CIEDE2000 formula). Mean and (standard deviation) are given. The perceptibility and acceptability thresholds [41] considered to describe the results were, respectively, 1.25 and 2.23 units.

	Groups (n=5)							
	FEL-G	**FEL-EG**	**LEU-G**	**LEU-EG**	**DIS-G**	**DIS-EG**	**ZLS-G**	**ZLS-EG**
ΔE_{00}	0.22 (0.04)^{b}	0.31 (0.04)^{a}	0.20 (0.04)^{b}	0.47 (0.11)^{a}	0.81 (0.04)^{b}	1.76 (0.05)^{a}	0.86 (0.09)^{b}	3.22 (0.22)^{a}

Different superscript letters denote significant differences ($P \leq 0.05$). Statistical comparisons are restricted to each ceramic material.

4 ARTIGO 2 – EXTENDED GLAZE FIRING OPTIMIZES THE FLEXURAL FATIGUE STRENGTH OF HARD-MACHINED CERAMICS

Este artigo científico foi submetido ao periódico *The Journal of Prosthetic Dentistry, Elsevier*, ISSN: 0022-3913, Fator de impacto = 2.095; Qualis A1. As normas para publicação estão descritas no Anexo B.

Extended glaze firing optimizes the flexural fatigue strength of hard-machined ceramics

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ABSTRACT

Statement of problem. Promising results showed that extended glaze firing induced greater crack healing than conventional (manufacturer-recommended) glaze firing, promoted tolerable residual stresses and produced clinically acceptable color alterations, without modifying the microstructure of densely sintered feldspathic, leucite- and lithium disilicate-based ceramics (Aurélio IL, Dorneles LS, May LG. Extended glaze firing on ceramics for hard machining: Crack healing, residual stresses, optical and microstructural aspects. Dent Mater 2017; 33(2):226-40). Moved by these previous findings and considering the processing damage associated to densely sintered computer-aided design, computer-aided manufacturing (CAD-CAM) ceramics, it is assumed that the ability of the extended glaze firing to improve the mechanical performance of these materials should be investigated.

Purpose. To analyze the effect of extended glaze firing (EG) on the flexural fatigue strength (FFS) of hard-machined leucite- (LEU) and lithium disilicate- (DIS) based ceramics.

Material and methods. Discs were machined from ceramic blocks and divided into six groups (n=20) according to the material, either LEU or DIS, and to the applied glaze firing - G, EG, or C (control/no heat treatment). The surface roughness data before/after firing were compared by paired-sample tests. Mean and standard deviations of FFS were calculated from staircase (up-and-down) tests (piston-on-three-ball, 500,000 cycles at 20 Hz) and analyzed by one-way ANOVA and *post-hoc* Tukey's test (α = .05).

Results. The surface roughness did not change after the firings (P >.05). The highest FFS in both ceramics was obtained after EG (LEU-EG = 80.52 ±6.3 MPa; DIS-EG = 147.25 ±10.5 MPa), which was statistically superior ($P \leq .05$) to G (LEU-G = 73 ±6.8 MPa; DIS-G = 134.34 ±15.6 MPa) and C (LEU-C = 61.94 ±6.3 MPa; DIS-C = 134.13 ±17.3 MPa).

Conclusions. Extended glaze firing optimizes the biaxial flexural fatigue strength of hard-

machined leucite- and lithium disilicate-based ceramics when compared to conventional glaze firing.

CLINICAL IMPLICATIONS

Based on the clinical implications that ceramic fracture represents, and considering the processing damage associated to densely sintered ceramics, the extended glaze firing should be considered as an alternative finishing protocol for these materials, mainly, for those with a higher vitreous content and, consequently, lower nominal strength.

INTRODUCTION

Critical flaws in ceramic materials can be introduced as a function of the processing method, which includes all the steps used in making a ceramic part, from shaping to firing. [1] Common flaws include sub-surface and surface damage from Computer Aided Design-Computer Aided Machining (CAD-CAM) and rotatory diamond grinding during internal adjustments. [2] Recently, Curran et al [3] assessed the surface/subsurface damage on ground feldspar and glass-ceramics for “hard machining”, [4] in which fully sintered blocks are milled into the expected restoration design, and estimated the potential losses in the strength, based on crack size measurements of the generated damage. Based on the results, which suggest a potential strength loss estimated between 33% and 54%, the authors [3] advise to carefully examine under a stereomicroscope the margin quality and the type of chip damage present in hard-machined ceramic blocks. Also, fractographic analysis of clinically failed all-ceramic restorations [5-9] showed that the fractures initiated from the cementation surface and/or the cervical margins, and consequently, procedures that affect these sites must be investigated regarding their influence on the ceramic strength.

Hard-machined restorations can be obtained from leucite- and lithium disilicate-based ceramics. [4,10] Favorable lifetime predictions indicated that 90% of milled leucite inlays/onlays and lithium disilicate single crowns would survive at least, 10.9 years and 20.9 years,

respectively. [11] However, current in vitro studies suggest that hard machining reduces the ceramic biaxial flexural strength, [10,12] as it introduces defects on the ceramic surface, and some clinical investigations still consider the ceramic fracture as one of the principal technical complications. [13-17] Thus, additional research is necessary to reduce ceramic fracture, considering the clinical implications associated with restoration replacement or repair. [1]

When it is intent to optimize the long-term performance of ceramic restorations, the effect of finishing procedures involving heat treatment should be taken into account. According to Denry, [1] manufacturer-recommended finishing procedures such as glazing seem to be inefficient to attenuate large flaws produced at the milling stage that may cause the fracture of the restorations. Also, conventional (manufacturer-recommended) glaze firing appears to significantly reduce the flexural strength of hard-machined glass-ceramics. [10,12] Thus, the viability of employing alternative glaze firings of densely sintered ceramics for CAD-CAM systems should be evaluated. [18]

Extended glaze firing [12,18] proposed by Aurélio et al [12] significantly increased the leucite flexural strength after machining when compared to the conventional glaze firing. Further results from Aurélio et al [18] demonstrated that crack healing was greater in densely sintered feldspathic, leucite-, and lithium disilicate-based ceramics specimens submitted to extended glaze firing than in those subjected to conventional (manufacturer-recommended) glaze firing. The authors [18] also reported tolerable residual stresses, clinically acceptable color alterations, and microstructural stability of these materials after the extended glaze regimen.

Clinically, ceramic restorations fail under fatigue, [19,20] when repetitive cyclic stresses of intensities below the material's normal strength are applied. [21-23] Since extended glaze firing showed promising results, [12,18] the influence of such alternative thermal treatments on the mechanical behavior of machined ceramics under fatigue should be investigated. The present study is intended to evaluate the effect of the extended glaze firing [12,18] on the biaxial

flexural fatigue strength of leucite- and lithium disilicate-based glass-ceramics. The hypothesis is that the extended glaze firing would improve the flexural fatigue strength of the specimens when compared to the conventional glaze firing and to no glaze firing (control) treatments.

Some studies [24,25] reported that ceramic surface roughness seems to be not affected by heat treatment. For Aurélio et al, [12] despite statistical significant, the reduction of surface roughness after the extended glaze firing had not influenced strength. However, crack propagation at lower levels of strength may initiate from areas in which surface roughness produces high stress concentration, particularly if the grooves are perpendicular to the tensile axis. [26,27] Therefore, as a complementary analysis, the influence of glaze firing treatments on the ceramic surface roughness was analyzed, and the tested hypothesis was that it would be reduced after the thermal treatment.

MATERIALS AND METHODS

One hundred and twenty discs (13.5 mm diameter, 1.4 mm thickness; ISO 6872:2008) [28] from fully sintered blocks (Table 1) were obtained by automated machining [10,12], using a CEREC inLab MC XL milling unit (Sirona Dental Systems Gmbh) - 60 discs of leucite "LEU" (IPS Empress CAD C14L, 14 mm × 14 mm × 18 mm, Ivoclar Vivadent AG), and 60 discs of a lithium disilicate "DIS" (IPS e.max CAD C14, 12.4 mm × 14.5 mm × 18 mm, Ivoclar Vivadent AG) glass-ceramic.

After milling, polishing was manually performed, using 400-, 600-, and 1,200-grit silicon carbide papers (Norton/Saint-Gobain Abrasivos Ltd.) on the upper surfaces of the discs in order to flatten the surface and achieve a thickness of 1.4 mm ±0.05 mm. The thickness was measured in the center of each specimen with a digital micrometer (Starret 210 MAP). The final crystallization stage of the lithium disilicate disc specimens was induced by a heat treatment at 850 °C for 25 min in a VACUMAT 6000 MP furnace (Vita Zahnfabrik).

Later, the surface roughness of the bottom sides of the discs, which were kept unchanged (as-machined), was measured. Discs from each ceramic material were randomly distributed among three experimental groups (n = 20 in each group), using the program Random Allocation (Version 1.0 - developed by M. Saghaei, Department of Anesthesia, Isfahan University of Medical Sciences, Isfahan, Iran). Statistical analysis was performed in order to validate the randomization, and the initial roughness was found to be similar among the groups ($P > .05$ in Kruskal-Wallis test).

Experimental groups and firing protocols are described in Table 2. Conventional (manufacturer-recommend) (G) and extended (EG) glaze firings were executed in a VITA VACUMAT 6000 MP furnace (Vita Zahnfabrik). As described in previous studies, [12,18] the thermal cycle for conventional glaze rigorously followed the manufacturer's recommendations. The same initial temperature, pre-heating time, and temperature increase rate from G regimen was adopted for the extended glaze firing. However, in EG the dwell time was set to 15 minutes, and slow cooling was performed by keeping the furnace closed until it reached 200 °C. As-machined control specimens (C) were not submitted to any firing.

The roughness of the bottom (machined) surface of the discs was measured before and after firing, using a profilometer with a contact-type stylus (SJ-410, Mitutoyo). Within each firing group, the treatment stage "before" (initial roughness of the discs, before they undergo heat treatment) served as control for the treatment stage "after" (final roughness of the discs, after they undergo heat treatment). [12]

The R_a (average surface roughness, μm) and R_z (arithmetic mean of the five highest peak-to-valley heights, μm) values [29] were determined using the average of three measurements on a path transverse to the machining direction. [10,12] The roughness measurement protocol was defined in ISO 4287:1997. [30]

A piston-on-three ball device was used for the biaxial flexural fatigue test (Instron ElectroPuls E3000, Instron Corporation), in the presence of water. A flat circular tungsten piston (Ø=1.6 mm) applied the load at the center of the discs. Specimens were positioned with the treated surface facing down (tensile stress) on the three support balls (Ø=3.2 mm), which were arranged 10 mm apart from each other in a triangular fashion (ISO 6872:2008). [28] An adhesive tape was fixed on the compression side of the discs before testing [31] in order to avoid the fragments from being spread [32] and to provide a more uniform contact between the piston and the sample. [33]

The flexural fatigue strength was determined for 500,000 cycles using the staircase (up-and-down) sensitivity test. [34] A sinusoidal load was applied, with the amplitude ranging from a minimum tensile stress of 15 MPa to the maximum tensile stress, at a frequency of 20 Hz (20 cycles per second). The maximum tensile stress applied to the first specimen (σ_1), equivalent to 50% of the mean monotonic flexural strength value, was 90 MPa for leucite and 138.50 MPa for lithium disilicate specimens; the step size, equivalent to 10% of σ_1, was 9 MPa for leucite and 13.85 MPa for lithium disilicate specimens. These values were determined based on the monotonic results from Aurélio et al [12] for the leucite ceramic (n=30, 180 MPa), and from a pilot study (n=5, piston-on-three-ball configuration, ISO 6872:2008, cross-head speed of 1 mm/min) for lithium disilicate specimens (277 MPa).

The first specimen of each group was then tested, and the next disc was submitted to a higher or lower tensile stress increment (step size value) depending on its survival or failure, respectively. The following equations were applied to calculate the load necessary to achieve the required maximum tensile stress:

$$\sigma = -0.2387P(X - Y)/d^2 \quad (1)$$

$$X = (1 + \nu)\ln(B/C)^2 + [(1 - \nu)/2](B/C)^2 \quad (2)$$

$$Y = (1 + \nu)[1 + \ln(A/C)^2] + (1 - \nu)(A/C)^2 \quad (3)$$

Where P is the load at fracture (N), d is the disc thickness (mm), v is the Poisson's ratio (0.25), A is the support ball radius (5 mm), B is the radius of the tip of the piston (0.8 mm), and C is the specimen radius (6.75 mm).

Load control was selected to interrupt the fatigue test at the moment of specimen failure, so that the number of cycles until failure could be recorded. The mean flexural fatigue strength (σ_f) and the standard deviation (s) of the groups were calculated considering all specimens from the point where the staircase plot acquired an up-and-down feature (reversal). [34]

After the mechanical tests, the specimens were analyzed using a light microscope (Stereo DiscoveryV20, Carl Zeiss, Göttingen), and then one disc from each evaluated group was explored under scanning electron microscopy (SEM, JSM-6360, JEOL) to identify the surface where the fracture initiated, according to the fractographic markings described by Quinn. [32]

The mean flexural fatigue strength (σ_f) and the standard deviation (s) were used for comparisons among the groups. ANOVA and a 95% post hoc test (Tukey's test, α = .05) were chosen for statistical analyses (STATA software, version 12, Stata Corp., College Station), based on the normality (P >.05, Shapiro-Wilk test) and homoscedasticity (P >.05, Bartlett's test) of the data and, on the statistical approaches being followed in current literature. [31,35-37] These approaches adopt more robust statistical tests for staircase sensitivity analysis in place of the maximum-likelihood estimation technique [38] which could be influenced by the standard deviation bias. [39-41]

The paired sample t-test at a 5% significance level was used to compare the mean surface roughness values (R_a, R_z), before (control) and after each glaze firing (intra-group analyses), when the data presented a normal distribution (P > .05, based on the Shapiro-Wilk

test). In the cases of non-normal distribution ($P \leq .05$, based on the Shapiro-Wilk test), Wilcoxon Signed Rank Test ($\alpha = .05$) was employed to analyze the mean surface roughness.

The sample size achieved a statistical power (STATA software, version 12, Stata Corp., College Station) higher than 0.8 (80%).

RESULTS

The results from the intra-group surface roughness comparisons, restricted to each firing protocol and ceramic material, are shown in Table 3. The surface roughness according to the *Ra* and *Rz* parameters of LEU and DIS specimens was not statistically modified ($P > .05$) after G and EG firings.

The mean biaxial flexural fatigue strength values (Table 3) and the patterns of runouts and failures of LEU and DIS for each experimental group are presented in Figures 1 and 2, respectively. The extended glaze firing group (EG) had the highest flexural fatigue strength for 500,000 mechanical cycles at 20 Hz, in both leucite (LEU-EG = 80.52 ±6.3 MPa) and lithium disilicate (DIS-EG = 147.25 ±10.5 MPa) specimens. The extended glaze firing significantly increased ≈9.5% the flexural fatigue strength values of leucite and lithium disilicate ceramics after machining when compared to conventional (manufacturer recommended) firing (LEU-G = 73 ±6.8 MPa; DIS-G = 134.34 ±15.6 MPa). In comparison to the control, this effect was most evident for a leucite (14.2%, LEU-C = 61.94 ±6.3 MPa) than for lithium disilicate (9%, DIS-C=134.13 ±17.3 MPa). The flexural fatigue strength of the leucite discs increased after the conventional glaze cycle when compared to the control specimens; however, no change was observed in the lithium disilicate ceramics.

A similar failure pattern was identified in the SEM images for all groups as seen in Figure 3, in which fractographic markings, such as Wallner lines, grinding cracks in Figures 3(c,d), 3(g,h), 3(i,j), and 3(k,l), and microstructural irregularities in Figures 3(a,b), 3(e,f) lead to the origin site on the tensile surface.

DISCUSSION

Extended glaze firing has shown promising results for glass-ceramics used for hard machining. This heat treatment significantly improved the flexural strength of milled leucite specimens, [12] had a greater ability to seal defects than the conventional glaze firing, developed tolerable residual stresses, and produced clinically acceptable color alterations, without changing the microstructure of feldspathic, leucite-, and lithium disilicate-based ceramics. [18] Since restoration failure happens throughout cyclic loading, [20,23] the influence of these alternative firings on the mechanical behavior of hard-machined leucite and lithium disilicate ceramics under fatigue was investigated in the present study. As a complementary analysis, the influence of glaze firing treatments on the ceramic surface roughness was also evaluated. CAD-CAM samples were used in order to simulate the heat treatment effects on restorations damaged by the machining process. [12] It was observed that the extended glaze firing improved the flexural fatigue strength of the leucite- and lithium disilicate-based ceramics, when compared to the conventional glaze firing and to no glaze firing (control). The first hypothesis was therefore accepted. However, since the surface roughness parameters of LEU and DIS specimens were not statistically different after EG and G firings, the second hypothesis was rejected.

Aurélio et al [18] reported that the extended glaze firing induced additional healing of artificially-created defects on the ceramic surface, in comparison with the conventional firing for leucite- and lithium disilicate-based specimens. So, the increase in the flexural fatigue strength in both of the tested materials after EG (Fig. 1 and 2) may be associated to some extent to crack modification process. The higher glass content of the leucite-based ceramic available for healing mechanisms, [42,43] in comparison to lithium disilicate, may explain not only why the increase in fatigue strength values after G was restricted to the LEU specimens, but also why the influence of EG on flexural fatigue strength was more evident in the leucite-

based ceramics. For LEU, the flexural fatigue strength after EG increased by 14.2% when compared to the control, whereas for DIS the increase was around 9%. Although EG mechanically benefited both LEU and DIS ceramics, the improvement of flexural fatigue strength values achieved after this firing may be more clinically relevant for LEU, since it presents lower nominal strength if compared to DIS [44].

It was shown that G firing improves the flexural fatigue strength of leucite hard-machined specimens if compared to no firing. However, some previous investigations [10,12] reported a decrease in monotonic flexural strength after G for identical specimens. These divergent results may be explained due to the different loading set-ups adopted by the studies (cyclic versus static loading). Crack initiation and growth that occurs under cyclic loading, comprising microcracking ahead of the propagating cracks, and may not happen under static loading. [45]

Surface roughness complementary analysis was performed before and after firing in an attempt to investigate the possible influence of this variable on flexural fatigue strength. Roughness may induce the formation of high stress concentration zones, particularly if the grooves are perpendicular to the tensile axis, which could lead to flaw propagation at lower levels of strength. [26,27] In the studies conducted by Addison et al [24] and Hung et al [25] ceramic surface roughness was not affected by heat treatment. Nevertheless, crack healing and an improvement in the strength (233.7 to 276.1 MPa) were detected after glaze firing by Hung et al [25] on lithium disilicate ceramic discs previously subjected to grinding with diamond burs. Our findings demonstrated that even though there were no significant changes in the ceramic roughness, the fatigue strength was improved after extended glaze firing probably owing to the ability of EG treatment to heal the defects. [18] Probably, surface roughness has less effect on strength, if compared to machining microcracks. [12] Also, the surface roughness analysis may be not sensitive enough to detect the changes in the ceramic, such as those caused by the

application of heat treatment. De Jager et al [27] argues that the tip of the profilometer could hardly perceive microcracks present on the ceramic surface since it is larger than the regular crack size.

Fractographic markings [32] such as the concave side of the Wallner lines, surface-distributed grinding cracks, and microstructural irregularities, helped to recognize the possible origin of fracture in the samples. As expected, in all specimens, the failures seemed to be initiated near or on the surface subjected to tensile stress during the biaxial flexural test. Therefore, the fractures did not occur due to high stresses at other possible sites, such as the contact surface between the disc and the piston [20] that could invalidate the flexural strength results.

Based on the clinical implications that ceramic fracture represents [1] (discomfort for the patient and embarrassment for the dentist, additional clinical time for procedures repetition, and profits reduction), any laboratory step capable to reduce the risk of failure in the restorations should be considered. Alternative treatments such as the extended glaze firing [12,18], which benefit the performance of densely sintered glass-ceramics for CAD-CAM systems and compensate other possible processing damages, deserve detailed attention in further studies. Additional approaches aiming to better simulate the clinical conditions should be performed. Since specimen geometry plays a crucial role in the fatigue behavior, crown-shaped specimens cemented onto standardized tooth models are recommended in future investigations.

CONCLUSIONS

Extended glaze firing improves the biaxial flexural fatigue strength of hard-machined leucite- and lithium disilicate-based ceramics when compared to conventional (manufacturer-recommended) glaze firing. Since it has been demonstrated in recent literature [12,18] to have positive effects on the mechanical properties, with clinically acceptable color alterations, it

could be considered as an alternative to finishing restorations made from these materials, mainly, for those with a higher vitreous content and, consequently, lower nominal strength.

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FIGURES

Fig. 1. Staircase sensitivity test results - mean biaxial flexural fatigue strength values (represented by horizontal lines) and standard deviation - during mechanical cycling (500,000 cycles at 20 Hz) for leucite-based ceramic discs (n=20): (a) after glaze firing, (b) after extended glaze firing, and (c) as-machined (control). Mean and standard deviation values were calculated considering all tested specimens from point where first reversal occurred ("Start point": G, n=16; EG, n=19, and C, n=17). Different superscripts and capital letters indicate groups with statistically significant differences ($P \leq .05$ in Tukey's test).

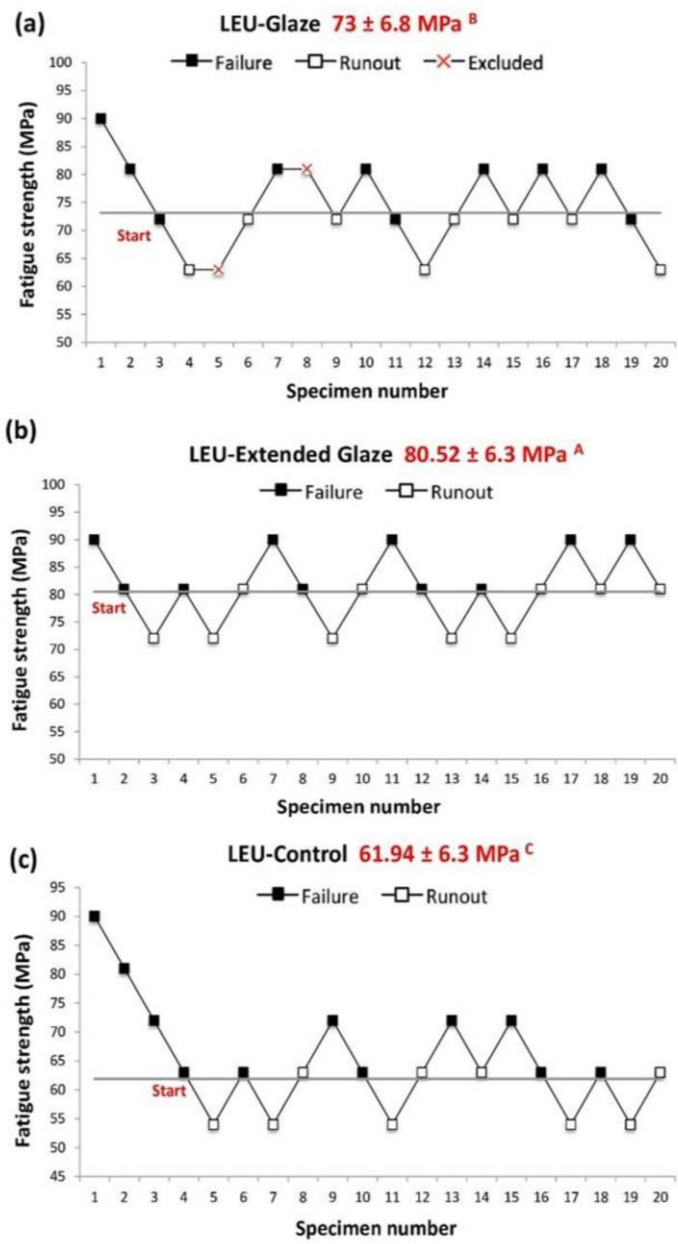


Fig. 2. Similar to Figure 1, except: Staircase sensitivity test results for lithium disilicate-based ceramic discs. Mean and standard deviation values were calculated considering all tested specimens from point where first reversal occurred ("Start point": G, n=20; EG, n=19, and C, n=19).

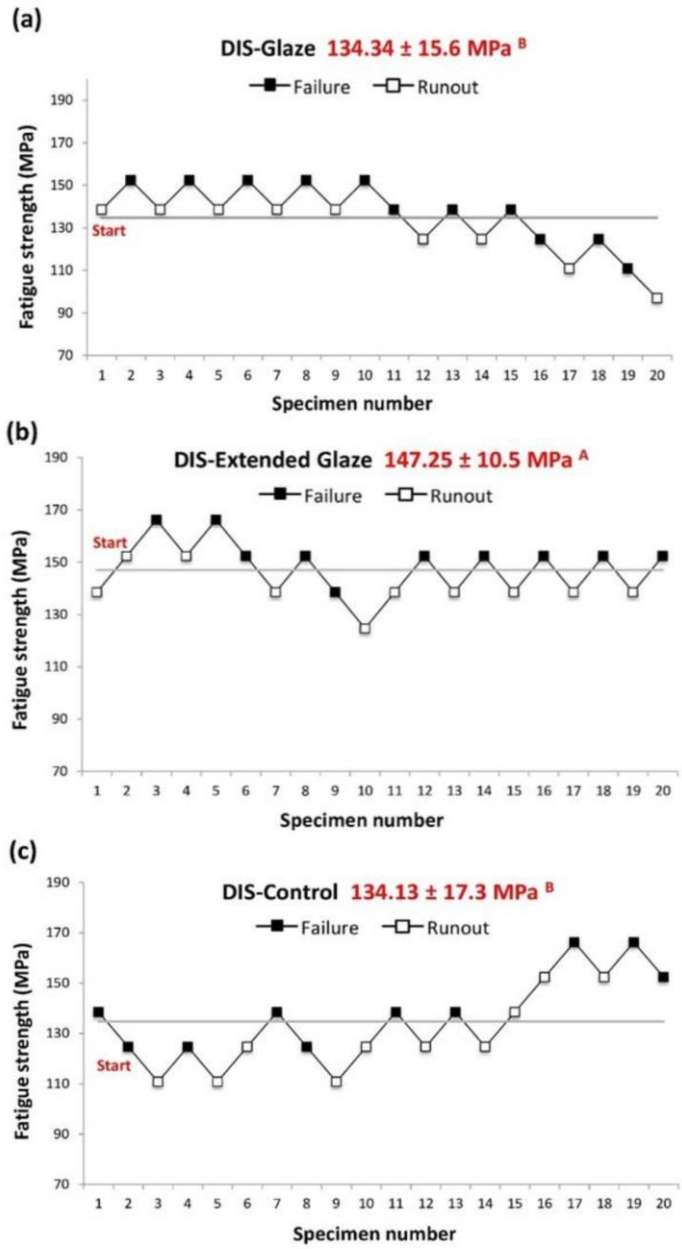


Fig. 3. Representative SEM images (100x and 200x) of fracture surface of LEU-G (a, b), LEU-EG (c, d), LEU-C (e, f), DIS-G (g, h), DIS-EG (i, j), and DIS-C (k, l) groups. Fractographic marks lead to bottom surface (tensile surface): a) in all specimens, formed primary Wallner lines (WL; white dashed curves), which are perpendicular to direction of crack propagation (DCP; large arrows), run to both sides of specimen due to biaxial bending stresses enclosing potential origin site (multiple thin arrows); b) surface-distributed grinding cracks in LEU-EG (c, d), DIS-G (g, h), DIS-EG (i, j), and DIS-C (k, l), and microstructural irregularities in LEU-G (a, b), and LEU-C (e, f) also indicate possible fracture origin in samples.

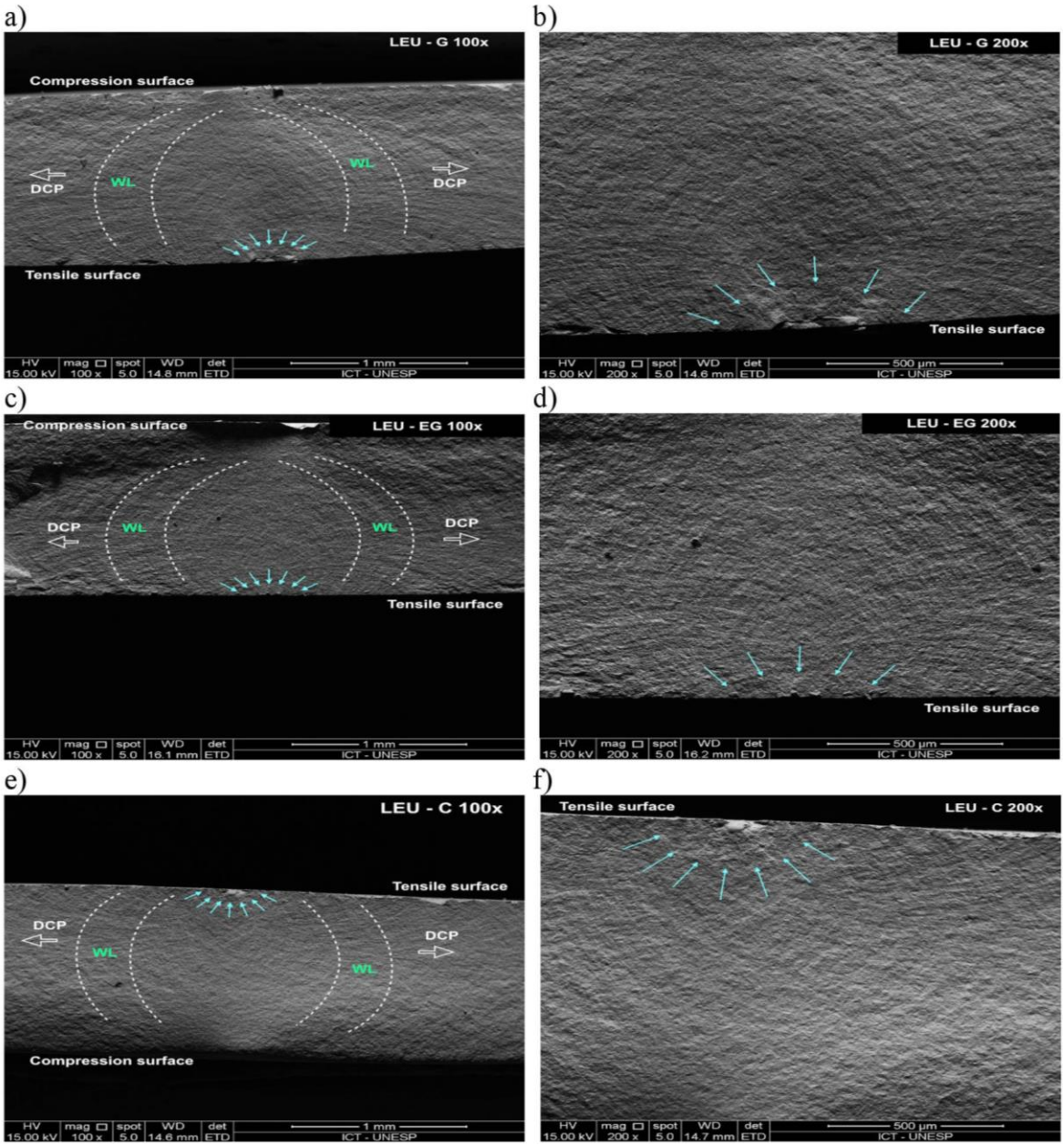
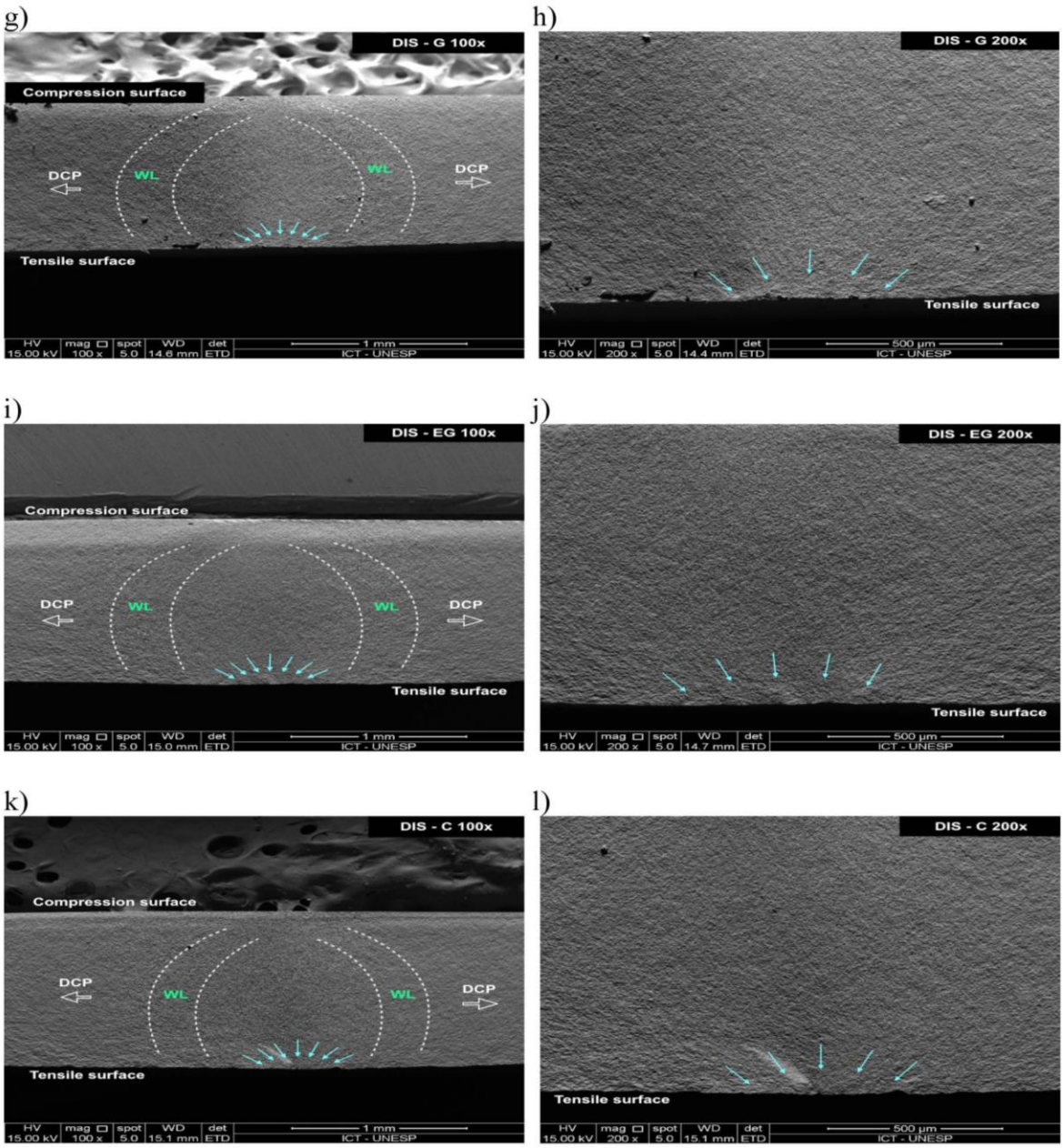


Fig. 3. (Continued).



TABLES

Table 1 – Ceramics for hard machining used in the study.

Ceramic	Code	Manufacturer	Microstructure	Composition[a]	Indications[a]
IPS Empress CAD	LEU	Ivoclar-Vivadent, Schaan, Liechtenstein	Leucite glass ceramic. Leucite crystals ($K_2O.Al_2O_3.4SiO_2$) ≈35-45%vol surrounded by a glassy matrix	Al_2O_3; SiO_2; K_2O; Na_2O; other oxides; pigments	Inlays, onlays, veneers, partial crowns, anterior and posterior single crowns
IPS e.max CAD (*Monolithic Solutions*)	DIS	Ivoclar-Vivadent, Schaan, Liechtenstein	Lithium disilicate glass ceramic. When fully crystallized: lithium disilicate crystals ($Li_2Si_2O_5$) ≈70%vol surrounded by a glassy matrix	SiO_2 ; Li_2O; K_2O; P_2O_5; ZrO_2; ZnO; Al_2O_3; MgO; other oxides	Inlays, onlays, veneers, anterior and posterior single crowns, and three-unit bridges (up to the second premolar as the terminal abutment). Single abutments and single abutment crowns for anterior and posterior teeth. CAD/CAM veneering technique

[a] Chemical composition and indications are described according to the manufacturers information.

Table 2. Description of experimental groups according to firing treatments applied to ceramic materials.

	Groups (n=20)					
Group code	**LEU-G**	**DIS-G**	**LEU-EG**	**DIS-EG**	**LEU-C**	**DIS-C**
Ceramic	Leucite	Lithium Disilicate	Leucite	Lithium Disilicate	Leucite	Lithium Disilicate
Glaze firing	Conventional glaze firing (G)[a]		Extended glaze firing (EG) [12,18]		No heat treatment; as-machined (Control)	
Initial temperature (°C)	403	403	403	403	-	
Temperature increase rate (°C/min)	100	60	100	60	-	
Final temperature (°C)	790	770	790	770	-	
Dwell time at final temperature (min)	1.5		15		-	
Vacuum	From 450 °C to 789 °C	From 450 °C to 769 °C	From 450 °C to 789 °C	From 450 °C to 769 °C	-	
Cooling rate	Fast: immediate furnace opening at end of dwell time		Slow: furnace remained closed until material reached 200 °C		-	

[a] Glaze firing protocol indicated for IPS Empress CAD and for IPS e.max CAD by manufacturer: Programat® - Firing programs tables. [46]

Table 3. Mean and standard deviation of surface roughness values (R_a and R_z), before (control) and after firing, and intra-group comparisons (*Paired samples t-test and ^Wilcoxon Signed Rank Test at 5% significance level).

Group	Treatment stage	R_a (µm) (Standard deviation)	*P*-value[1]	R_z (µm) (Standard deviation)	*P*-value[1]
LEU-G	Before	1.50(0.19)	.13*	9.15(1.23)	.26^
	After	1.53(0.21)		9.30(1.28)	
LEU-EG	Before	1.44(0.21)	.33^	8.98(1.25)	.23^
	After	1.40(0.24)		8.62(1.19)	
DIS-G	Before	1.70(0.23)	.88*	9.96(1.24)	.53^
	After	1.70(0.24)		9.83(1.21)	
DIS-EG	Before	1.66(0.19)	.53^	9.83(0.92)	.85*
	After	1.68(0.16)		9.86(0.93)	

[1] $P \leq .05$ indicates difference between groups.

5 DISCUSSÃO

Esforços constantes para reduzir as taxas de fratura de restaurações cerâmicas são necessários em decorrência das implicações clínicas e laboratoriais envolvidas neste tipo de falha, que ainda surge como complicação técnica em estudos clínicos envolvendo restaurações cerâmicas usinadas a partir de blocos densamente sinterizados (GUESS et al., 2013; OTTO; SCHNEIDER, 2008; PJETURSSON et al., 2015, 2017; REICH et al., 2014; SAILER et al., 2015; REISS; WALTHER, 2000). Tais fraturas e a redução dos valores de resistência cerâmica têm sido associadas à danos advindos de processos de desgaste (CURRAN et al., 2017), como a usinagem (FRAGA et al., 2015, 2017; KELLY et al., 1991; SCHERRER et al., 2011); que, por sua vez, parecem não ser efetivamente minimizados após o glazeamento das peças (DENRY, 2013).

Assim, a realização dos dois trabalhos laboratoriais que compõe esta tese foi impulsionada pela necessidade de estudos com ciclos térmicos alternativos, na busca por um tratamento de finalização que otimize o desempenho de cerâmicas densamente sinterizadas para usinagem em sistemas CAD-CAM e que seja factível na prática clínica e laboratorial. Diante disso, objetivou-se avaliar o emprego da queima estendida de glazeamento (GE) (AURÉLIO et al., 2015) como alternativa à queima convencional (G, sugerida pelo fabricante) para o acabamento destes materiais.

Esperou-se que maior seria o efeito do tratamento térmico sobre o comportamento mecânico dos materiais cerâmicos, quanto maior fosse o número de variáveis concomitantemente influenciadas por ele, desde que os efeitos produzidos não fossem antagônicos entre si. Assim, acreditou-se que a queima de glazeamento estendida (GE) poderia otimizar os valores de resistência flexural à fadiga dos materiais investigados, já que, de acordo com os achados do primeiro artigo, este ciclo conferiu maior selamento dos defeitos em relação ao ciclo convencional (G) e desenvolveu tensões residuais toleráveis; sem no entanto promover alterações ópticas além dos limiares clínicos e nem mudanças na microestrutura das cerâmicas feldspáticas, leucíticas, ou à base de dissilicato de lítio. A exceção deu-se apenas para a cerâmica à base de silicato de lítio reforçado por zircônia (SLZ), na qual a queima estendida GE promoveu variações de cor (ΔE_{00} = 3,22) e de translucidez (ΔCR = 0,12) que excederam a aceitabilidade clínica de ΔE_{00} = 2,23 (DELLA BONA et al., 2015) e ΔCR = 0,06 (LIU et al., 2010). Por este motivo, SLZ não pôde ser inserida na investigação do efeito de GE na resistência flexural à fadiga de cerâmicas de menor teor vítreo, descrita no segundo artigo desta tese.

Por questões de viabilidade – reduzir o tempo de execução e os custos do trabalho –, optou-se pelo uso exclusivo da leucita como material representante do grupo de cerâmicas com alto teor vítreo.

De fato, os espécimes usinados a partir de blocos cerâmicos de leucita e de dissilicato de lítio submetidos ao regime estendido (GE) atingiram valores de resistência flexural à fadiga estatisticamente superiores àqueles tratados com a queima convencional, e àqueles que não sofreram tratamento térmico (grupo controle). Tal efeito pareceu ser mais evidente na cerâmica leucítica, uma vez que a resistência flexural à fadiga após GE aumentou em torno de 14,2% em comparação ao grupo controle, enquanto que para o dissilicato de lítio esse aumento foi em torno de 9%. Ao que tudo indica, os processos de cicatrização via capilaridade (GIRARD; FAIVRE; DESPETIS, 2011; HRMA; HAN; COOPER, 1988) e oxidação (GREIL, 2012; QUEMARD et al., 2007) possivelmente tenham sido favorecidos pela estrutura com maior teor vítreo da leucita, se comparada a do dissilicato de lítio, com maior agregado cristalino. Aurélio e colaboradores (2015) já haviam demonstrado aumento significativo dos valores de resistência flexural de discos usinados em leucita após GE. Além disso, por se tratar de um material de menor resistência nominal em relação ao dissilicato de lítio (KELLY, 2004, 2008), a leucita talvez seja o material mais beneficiado clinicamente com a otimização dos valores de resistência flexural à fadiga alcançados por GE.

Contudo, estudos que busquem atenuar, de maneira factível na clínica/laboratório, os efeitos oriundos de danos promovidos no processamento de restaurações/espécimes à base de dissilicato de lítio densamente sinterizado e, consequentemente, otimizar os valores de resistência mecânica deste material, parecem não estar disponíveis. Pesquisas recentes sobre o efeito do acabamento via polimento, reglazeamento, ou associação de ambos no desempenho mecânico de espécimes de dissilicato de lítio usinados (MORES et al., 2017) ou seccionados em forma de barra (MOHAMMADIBASSIR et al., 2017) não incluíram nas comparações grupos controle “sem tratamento térmico”. Em quaisquer dos grupos estudados os espécimes já partiam de uma situação glazeada. Dessa forma, o efeito do tratamento térmico sobre a resistência da cerâmica processada (usinada, seccionada) não foi avaliado. Além do mais, os protocolos de glaze utilizados basearam-se no *overglaze*, que associa ao tratamento térmico a adição de pastas ou *sprays* de glaze, formando uma película sobre a superfície cerâmica. No entanto, é sabido que o processo de usinagem produz defeitos ao longo de todo o material (FRAGA et al., 2015; KELLY et al., 1991; SCHERRER et al., 2011), inclusive na face de cimentação, que sofre apenas a ação do tratamento térmico (CHEN et al., 2010).

Diferentemente, neste trabalho de tese, grupo controle “somente usinado/sem tratamento térmico” foi inserido para comparação e optou-se, além disso, por não utilizar o protocolo de *overglaze*. Assim, a repercussão do tratamento térmico (queima convencional e estendida de glazeamento) sobre o comportamento mecânico de espécimes de dissilicato de lítio que continham danos de usinagem (KELLY et al., 1991; SCHERRER et al., 2011) pôde ser avaliado isoladamente. Como resultado, a resistência flexural à fadiga do dissilicato de lítio aumentou significativamente após GE, se comparada aos valores grupo somente usinado (sem tratamento térmico). Diante disso, acredita-se que a queima GE merece ser considerada como uma alternativa de tratamento a ser estudada para o acabamento de restaurações densamente sinterizadas de dissilicato de lítio, mesmo que os efeitos de GE sobre este material tenham se mostrado aparentemente menos promissores quando comparados aos obtidos neste trabalho para a leucita.

Com relação às alterações de superfície, este trabalho indicou que a rugosidade dos discos usinados não foi modificada após as queimas G e GE. Dentre as as explicações plausíveis para tais achados supõem-se que o perfilômetro de contato não tenha sido sensível para detectar as mudanças na topografia de superfície (DE JAGER; FEILZER; DAVIDSON, 2000) ou ainda, que a alteração nos valores de resistência estejam vinculada às microtrincas promovidas pela usinagem, e não às alterações na rugosidade superficial (AURÉLIO et al., 2015).

Já, no que tange o desenvolvimento de tensões residuais, apesar do caráter oposto – enquanto o ciclo estendido de glazeamento promoveu tensões compressivas na superfície dos espécies, a queima convencional gerou tensões trativas –, ambas as queimas produziram tensões com magnitudes similares e níveis toleráveis (BENETTI et al., 2014), entre -12,19 MPa (compressão) e 14,46 MPa (tração), sugerindo que esta variável pareceu não interferir de maneira significativa no comportamento mecânico dos materiais investigados. Sob cargas cíclicas, o efeito da queima convencional de glazeamento na cicatrização de defeitos ao longo de toda a espessura (volume) dos espécimes pareceu superar qualquer possível prejuízo mecânico vinculado às tensões trativas detectadas na superfície dos materiais.

Por fim, de acordo com a fractografia, em todos os espécimes, as falhas pareceram ser iniciadas próximo ou na superfície sujeita às tensões de tração durante o teste de flexão biaxial, indicando que as fraturas não ocorreram devido à concentração de altas tensões em outras possíveis regiões, como a superfície de contato entre o disco e o pistão (KELLY et al., 2010). Além disso, foram descritos os tipos de origem, conforme sugerido por Scherrer et al. (2017) e, na quase totalidade dos espécies, foram reconhecidas origens do tipo “trincas de

desgaste distribuídas em superfície” *(“Surface-distributed grinding cracks”).* Dessa forma, os resultados da análise fractográfica vem ao encontro de outros achados que associam a fratura cerâmica à danos de usinagem (KELLY et al., 1991; SCHERRER et al., 2011) ou provenientes de algum outro tipo de desgaste (CURRAN et al., 2017).

Em função das limitações encontradas nos estudos que compõe esta tese, salienta-se que investigações adicionais com o objetivo de melhor simular as condições clínicas também devem ser conduzidas. Espécimes que reproduzam a anatomia da porção coronária cimentados a troqueis padronizados são recomendados em trabalhos futuros. Diante dos resultados promissores até o presente momento, o estudo da compatibilidade da queima estendida de glazeamento com as pastas ou *sprays* de glaze também se faz necessário.

6 CONCLUSÃO

A queima estendida de glazeamento conferiu maior selamento dos defeitos em relação ao ciclo convencional, estipulado pelo fabricante, desenvolvendo níveis toleráveis de tensão residual em cerâmicas feldspática, leucítica, à base de dissilicato de lítio e à base de silicato de lítio reforçado por zircônia densamente sinterizadas.

As cerâmicas feldspática, vítrea à base de leucita e vítrea à base de dissilicato de lítio mantiveram suas propriedades ópticas dentro do limiar clinicamente aceitável e não apresentaram alterações detectáveis na microestrutura após serem submetidos à queima estendida de glazeamento. A exceção deu-se para a cerâmica à base de silicato de lítio reforçado por zircônia que, após o tratamento térmico, apresentou-se opticamente instável, conferindo uma aparência estética inaceitável ao material.

Mesmo após a usinagem, a queima estendida de glazeamento foi capaz de otimizar em igual magnitude os valores de resistência flexural à fadiga de espécimes vitrocerâmicos reforçados por leucita, e à base de dissilicato de lítio se comparada ao ciclo convencional. No entanto, quando comparado à espécimes controle, somente usinados, o aumento da resistência à fadiga após a queima estendida foi mais significativo para a cerâmica leucítica do que para o dissilicato de lítio.

Assim, com base nos achados desta tese, conclui-se que a queima estendida de glazeamento apresenta-se como uma possível alternativa para o acabamento de restaurações feldspáticas, leucíticas e à base de dissilicato de lítio usinadas; em benefício, principalmente, daquelas de maior teor vítreo e, consequentemente, menor resistência nominal.

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ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *DENTAL MATERIALS*

GUIDE FOR AUTHORS

Authors are requested to submit their original manuscript and figures via the online submission and editorial system for Dental Materials. Using this online system, authors may submit manuscripts and track their progress through the system to publication. Reviewers can download manuscripts and submit their opinions to the editor. Editors can manage the whole submission/review/revise/publish process. Please register at: http://ees.elsevier.com/dema.

Dental Materials now only accepts online submissions.

The Artwork Quality Control Tool is now available to users of the online submission system. To help authors submit high-quality artwork early in the process, this tool checks the submitted artwork and other file types against the artwork requirements outlined in the Artwork Instructions to Authors on http://www.elsevier.com/artworkinstructions. The Artwork Quality Control Tool automatically checks all artwork files when they are first uploaded. Each figure/file is checked only once, so further along in the process only new uploaded files will be checked.

Manuscripts

The journal is principally for publication of Original Research Reports, which should preferably investigate a defined hypothesis. Maximum length 6 journal pages (approximately 20 double-spaced typescript pages) including illustrations and tables.

Systematic Reviews will however be considered. Intending authors should communicate with the Editor beforehand, by email, outlining the proposed scope of the review. Maximum length 10 journal pages (approximately 33 double-spaced typescript pages) including figures and tables. Three copies of the manuscript should be submitted: each accompanied by a set of illustrations. The requirements for submission are in accordance with the "Uniform Requirements for Manuscripts Submitted to Biomedical Journals", Annals of Internal Medicine, 1997,126, 36-47. All manuscripts must be written in American English. Authors are urged to write as concisely as possible. The Editor and Publisher reserve the right to make minimal literary corrections for the sake of clarity. Authors for whom English is not the first language should have their manuscripts read by colleagues fluent in English. If extensive English corrections are needed, authors may be charged for the cost of editing. For additional reference, consult issues of Dental Materials published after January 1999 or the Council of Biology Editors Style Manual (1995 ed.).

All manuscripts should be accompanied by a letter of transmittal, signed by each author, and stating that the manuscript is not concurrently under consideration for publication in another journal, that all of the named authors were involved in the work leading to the publication of the paper, and that all the named authors have read the paper before it is submitted for publication. Always keep a backup copy of the electronic file for reference and safety. Manuscripts not conforming to the journal style will be returned. In addition, manuscripts, which are not written in fluent English, will be rejected automatically without refereeing.

Article structure

Subdivision - numbered sections

Divide your article into clearly defined and numbered sections. Subsections should be numbered 1.1 (then 1.1.1, 1.1.2, ...), 1.2, etc. (the abstract is not included in section numbering). Use this numbering also for internal cross-referencing: do not just refer to 'the text'. Any subsection may be given a brief heading. Each heading should appear on its own separate line.

Introduction

This must be presented in a structured format, covering the following subjects, although actual subheadings should not be included:

• succinct statements of the issue in question;
• the essence of existing knowledge and understanding pertinent to the issue (reference);
• the aims and objectives of the research being reported relating the research to dentistry, where not obvious.

Materials and methods

• describe the procedures and analytical techniques.
• only cite references to published methods.
• include at least general composition details and batch numbers for all materials.
• identify names and sources of all commercial products e.g.

"The composite (Silar, 3M Co., St. Paul, MN, USA)..."
"... an Au-Pd alloy (Estheticor Opal, Cendres et Metaux, Switzerland)."

• specify statistical significance test methods.
Results
• refer to appropriate tables and figures.
• refrain from subjective comments.
• make no reference to previous literature.
• report statistical findings.

Discussion

• explain and interpret data.
• state implications of the results, relate to composition.
• indicate limitations of findings.
• relate to other relevant research.

Conclusion (if included)

• must NOT repeat Results or Discussion
• must concisely state inference, significance, or consequences

Appendices

If there is more than one appendix, they should be identified as A, B, etc. Formulae and equations in appendices should be given separate numbering: Eq. (A.1), Eq. (A.2), etc.; in a subsequent appendix, Eq. (B.1) and so on. Similarly for tables and figures: Table A.1; Fig. A.1, etc.

Essential title page information

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• Author names and affiliations. Please clearly indicate the given name(s) and family name(s) of each author and check that all names are accurately spelled. Present the authors' affiliation addresses (where the actual work was done) below the names. Indicate all affiliations with a lowercase superscript letter immediately after the author's name and in front of the appropriate address.
Provide the full postal address of each affiliation, including the country name and, if available, the e-mail address of each author.
• Corresponding author. Clearly indicate who will handle correspondence at all stages of refereeing and publication, also post-publication. Ensure that the e-mail address is given and that contact details are kept up to date by the corresponding author.
• Present/permanent address. If an author has moved since the work described in the article was done, or was visiting at the time, a 'Present address' (or 'Permanent address') may be indicated as a footnote to that author's name. The address at which the author actually did the work must be retained as the main, affiliation address. Superscript Arabic numerals are used for such footnotes.

Abstract (structured format)

• 250 words or less.
• subheadings should appear in the text of the abstract as follows: Objectives, Methods, Results, Significance. (For Systematic Reviews: Objectives, Data, Sources, Study selection, Conclusions). The Results section may incorporate small tabulations of data, normally 3 rows maximum.

Keywords

Up to 10 keywords should be supplied e.g. dental material, composite resin, adhesion.

Abbreviations

Define abbreviations that are not standard in this field in a footnote to be placed on the first page of the article. Such abbreviations that are unavoidable in the abstract must be defined at their first mention there, as well as in the footnote. Ensure consistency of abbreviations throughout the article.

Acknowledgements

Collate acknowledgements in a separate section at the end of the article before the references and do not, therefore, include them on the title page, as a footnote to the title or otherwise. List here those individuals who provided help during the research (e.g., providing language help, writing assistance or proof reading the article, etc.).

Units

Follow internationally accepted rules and conventions: use the international system of units (SI). If other units are mentioned, please give their equivalent in SI.

Math formulae

Please submit math equations as editable text and not as images. Present simple formulae in line with normal text where possible and use the solidus (/) instead of a horizontal line for small fractional terms, e.g., X/Y. In principle, variables are to be presented in italics. Powers of e are often more conveniently denoted by exp. Number consecutively any equations that have to be displayed separately from the text (if referred to explicitly in the text).

Embedded math equations

If you are submitting an article prepared with Microsoft Word containing embedded math equations then please read this related support information (http://support.elsevier.com/app/answers/detail/a_id/302/).

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• Supply files that are too low in resolution;
• Submit graphics that are disproportionately large for the content.

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• should be complete and understandable apart from the text.
• include key for symbols or abbreviations used in Figures.
• individual teeth should be identified using the FDI two-digit system.

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Moulin P, Picard B and Degrange M. Water resistance of resin-bonded joints with time related to alloy surface treatments. J Dent, 1999; 27:79-87. 2. Taylor DF, Bayne SC, Sturdevant JR and Wilder AD. Comparison of direct and indirect methods for analyzing wear of posterior composite restorations. Dent Mater, 1989; 5:157-160. Avoid referencing abstracts if possible. If unavoidable, reference as follows: 3. Demarest VA and Greener EH . Storage moduli and interaction parameters of experimental dental composites. J Dent Res, 1996; 67:221, Abstr. No. 868.

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Text: Indicate references by number(s) in square brackets in line with the text. The actual authors can be referred to, but the reference number(s) must always be given. Example: '..... as demonstrated [3,6]. Barnaby and Jones [8] obtained a different result ....' List: Number the references (numbers in square brackets) in the list in the order in which they appear in the text.

Examples:

Reference to a journal publication:

[1] J. van der Geer, J.A.J. Hanraads, R.A. Lupton, The art of writing a scientific article, J. Sci. Commun. 163 (2010) 51–59.

Reference to a book:

[2] W. Strunk Jr., E.B. White, The Elements of Style, fourth ed., Longman, New York, 2000.

Reference to a chapter in an edited book:

[3] G.R. Mettam, L.B. Adams, How to prepare an electronic version of your article, in: B.S. Jones, R.Z. Smith (Eds.), Introduction to the Electronic Age, E-Publishing Inc., New York, 2009, pp. 281–304.

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ANEXO B – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO *THE JOURNAL PROSTHETIC DENTISTRY*

Article Types

Articles are classified as one of the following: research/clinical science article, clinical report, technique article, systematic review, or tip from our readers. Required sections for each type of article are listed in the order in which they should be presented.

Research and Education/Clinical Research

The research report should be no longer than 10-12 double-spaced, typed pages and be accompanied by no more than 12 high-quality illustrations. Avoid the use of outline form (numbered and/or bulleted sentences or paragraphs). The text should be written in complete sentences and paragraph form.

Abstract (approximately 400 words): Create a structured abstract with the following subsections: Statement of Problem, Purpose, Material and Methods, Results, and Conclusions. The abstract should contain enough detail to describe the experimental design and variables. Sample size, controls, method of measurement, standardization, examiner reliability, and statistical method used with associated level of significance should be described in the Material and Methods section. Actual values should be provided in the Results section.

Clinical Implications: In 2-4 sentences, describe the impact of the study results on clinical practice.

Introduction: Explain the problem completely and accurately. Summarize relevant literature, and identify any bias in previous studies. Clearly state the objective of the study and the research hypothesis at the end of the Introduction. Please note that, for a thorough review of the literature, most (if not all references) should first be cited in the Introduction and/or Material and Methods section.

Material and Methods: In the initial paragraph, provide an overview of the experiment. Provide complete manufacturing information for all products and instruments used, either in parentheses or in a table. Describe what was measured, how it was measured, and the units of measure. List criteria for quantitative judgment. Describe the experimental design and variables, including defined criteria to control variables, standardization of testing, allocation of specimens/subjects to groups (specify method of randomization), total sample size, controls, calibration of examiners, and reliability of instruments and examiners. State how sample sizes were determined (such as with power analysis). Avoid the use of group numbers to indicate groups. Instead, use codes or abbreviations that will more clearly indicate the characteristics of the groups and will therefore be more meaningful for the reader. Statistical tests and associated significance levels should be described at the end of this section.

Results: Report the results accurately and briefly, in the same order as the testing was described in the Material and Methods section. For extensive listings, present data in tabular or graphic form to help the reader. For a 1-way ANOVA report of, F and *P* values in the appropriate location in the text. For all other ANOVAs, per guidelines, provide the ANOVA table(s). Describe the most significant findings and trends. Text, tables, and figures should not repeat each other. Results noted as significant must be validated by actual data and P values.

Discussion: Discuss the results of the study in relation to the hypothesis and to relevant literature. The Discussion section should begin by stating whether or not the data support rejecting the stated null hypothesis. If the results do not agree with other studies and/or with accepted opinions, state how and why the results differ. Agreement with other studies should also be stated. Identify the limitations of the present study and suggest areas for future research.

Conclusions: Concisely list conclusions that may be drawn from the research; do not simply restate the results. The conclusions must be pertinent to the objectives and justified by the data. In most situations, the conclusions are true for only the population of the experiment. All statements reported as conclusions should be accompanied by statistical analyses.
*References:*See Reference Guidelines and Sample References page.

Tables: See Table Guidelines.

Illustrations: See Figure Submission and Sample Figures page.

Clinical Report

The clinical report describes the author's methods for meeting a patient treatment challenge. It should be no longer than 4 to 5 double-spaced, pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the problem encountered and treatment administered.

Introduction: Summarize literature relevant to the problem encountered. Include references to standard treatments and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Clinical Report section.

Clinical Report: Describe the patient, the problem with which he/she presented, and any relevant medical or dental background. Describe the various treatment options and the reasons for selection of the chosen treatment. Fully describe the treatment rendered, the length of the follow-up period, and any improvements noted as a result of treatment. This section should be written in past tense and in paragraph form.

Discussion: Comment on the advantages and disadvantages of the chosen treatment and describe any contraindications for it. If the text will only be repetitive of previous sections, omit the Discussion.

Summary: Briefly summarize the patient treatment.

References: See Reference Guidelines and Sample References page.

Illustrations: See Figure Submission and Sample Figures page.

Dental Technique

The dental technique article presents, in a step-by-step format, a unique procedure helpful to dental professionals. It should be no longer than 4 to 5 double-spaced, typed pages and be accompanied by no more than 8 high-quality illustrations. In some situations, the Editor may approve the publication of additional figures if they contribute significantly to the manuscript.

Abstract: Provide a short, nonstructured, 1-paragraph abstract that briefly summarizes the technique.

Introduction: Summarize relevant literature. Include references to standard methods and protocols. Please note that most, if not all, references should first be cited in the Introduction and/or Technique section.

Technique: In a numbered, step-by-step format, describe each step of the technique. The text should be written in command rather than descriptive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed.") Include citations for the accompanying illustrations.

Discussion: Comment on the advantages and disadvantages of the technique, indicate the situations to which it may be applied, and describe any contraindications for its use. Avoid excessive claims of effectiveness. If the text will only be repetitive of previous sections, omit the Discussion.

Summary: Briefly summarize the technique presented and its chief advantages.

References: See Reference Guidelines and Sample References page

Illustrations: See Figure Submission and Sample Figures page.

Systematic Review

The author is advised to develop a systematic review in the Cochrane style and format. The *Journal* has transitioned away from literature reviews to systematic reviews. For more information on systematic reviews,

please see www.cochrane.org. An example of a Journal systematic review: Torabinejad M, Anderson P, Bader J, Brown LJ, Chen LH, Goodacre CJ, Kattadiyil MT, Kutsenko D, Lozada J, Patel R, Petersen F, Puterman I, White SN. Outcomes of root canal treatment and restoration, implant-supported single crowns, fixed partial dentures, and extraction without replacement: a systematic review. J Prosthet Dent 2007;98:285-311.

The systematic review consists of:

An Abstract using a structured format (Statement of Problem, Purpose, Material and Methods, Results, Conclusions).

Text of the review consisting of an introduction (background and objective), methods (selection criteria, search methods, data collection and data analysis), results (description of studies, methodological quality, and results of analyses), discussion, authors' conclusions, acknowledgments, and conflicts of interest. References should be peer reviewed and follow JPD format.

Tables and figures, if necessary, showing characteristics of the included studies, specification of the interventions that were compared, the results of the included studies, a log of the studies that were excluded, and additional tables and figures relevant to the review.

Tips From Our Readers

Tips are brief reports on helpful or timesaving procedures. They should be limited to 2 authors, no longer than 250 words, and include no more than 2 high quality illustrations. Describe the procedure in a numbered, step-by-step format; write the text in command rather than descriptive or passive form ("Survey the diagnostic cast" rather than "The diagnostic cast is surveyed").

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The number of authors is limited to 4; the inclusion of more than 4 *must be justified* in the letter of submission. (Each author's contribution must be listed.) Otherwise, contributing authors in excess of 4 will be listed in the Acknowledgments. There can only be one corresponding author.

General Formatting
All submissions must be submitted via the EES system in Microsoft Word with an 8.5×11 inch page size. The following specifications should also be followed:
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- Headers/Footers should be clear of page numbers or other information
- Headings are upper case bold, and subheads are upper/lower case bold. No italics are used.
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