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Sara Fraga

COMPORTAMENTO À FADIGA DE MATERIAIS CERÂMICOS

Santa Maria, RS
2016

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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 título de **Doutor em Ciências Odontológicas**.

Orientadora: Profa. Dra. Liliana Gressler May

Santa Maria, RS
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Aprovado em 09 de dezembro de 2016:

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Liliana Gressler May, Dra. (UFSM)
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Santa Maria, RS
2016

DEDICATÓRIA

Aos meu pais, Nei e Zenaide,
todo o meu amor e a minha gratidão.

À professora Liliana,
meu carinho, respeito e admiração.

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RESUMO

COMPORTAMENTO À FADIGA DE MATERIAIS CERÂMICOS

AUTORA: SARA FRAGA
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A presente tese é composta por dois artigos científicos, cujo tema principal é fadiga em materiais cerâmicos. Artigo 1: Impacto da usinagem CAD/CAM no comportamento à fadiga de cerâmicas vítreas e policristalinas. Esse trabalho objetivou investigar o efeito da usinagem CAD/CAM na resistência à fadiga e na rugosidade superficial de cerâmicas vítreas e policristalinas, por meio da comparação entre corpos de prova usinados e corpos de prova totalmente polidos após a usinagem. Blocos de zirconia, dissilicato de lítio e cerâmica vítrea reforçada por leucita foram usinados em sistema CAD/CAM no formato de discos, e divididos em dois grupos: usinagem (U); e usinagem seguida de polimento (UP). A rugosidade superficial (Ra e Rz) foi avaliada em perfilômetro de contato e a resistência à fadiga flexural determinada pelo método *step-test* (n = 20), utilizando dispositivo de flexão biaxial *piston-on-three ball* (ISO 6872:2008). O protocolo de carregamento variou de acordo com o tipo de cerâmica, e foi estabelecido com base em ensaio monotônico prévio (n = 5). Dez mil ciclos foram aplicados em cada *step*, a uma frequência de 1,4 Hz. Estatística de Weibull foi utilizada para a análise dos dados de fadiga e teste não paramétrico Mann-Whitney (α = 0,05) para comparação dos valores de Ra e Rz entre as condições U e UP de cada material. Os resultados mostraram que a usinagem resultou em maior rugosidade superficial e menores valores de resistência característica à fadiga, quando comparada aos grupos totalmente polidos após a usinagem. Maior redução na resistência característica à fadiga de UP para U foi observada na zircônia (40%; U = 536,48 MPa; UP = 894,50 MPa), seguida pelo dissilicato de lítio (33%; U = 187,71 MPa; UP = 278,93 MPa) e leucita (29%; U = 72,61 MPa; UP = 102,55 MPa). Sendo assim, concluiu-se que a usinagem CAD/CAM afeta negativamente a resistência à fadiga flexural de cerâmicas com diferentes microestruturas. Os resultados sugerem que a usinagem de materiais parcialmente sinterizados pode ser tão deletéria quanto a usinagem de materiais totalmente sinterizados. Artigo 2: Frequências de até 20 Hz como uma alternativa para acelerar ensaios de resistência à fadiga cíclica em cerâmica Y-TZP. Esse trabalho objetivou investigar a influência da frequência de aplicação de carga na resistência à fadiga da zircônia, utilizando-se o método da escada, com número máximo de ciclos fixado em 500.000, e configuração de ensaio *piston-on-three ball* (ISO 6872:2008). As frequências investigadas foram 2 Hz (controle – simulação da atividade mastigatória; n = 20), 10 Hz (n = 20), 20 Hz (n = 20) e 40 Hz (n = 21). Os resultados foram submetidos a análise de variância e teste de Tukey (α = 0,05). A resistência à fadiga foi significativamente superior para o grupo 40 Hz (630,7 MPa) e não diferiu entre os grupos 2 Hz (550,3 MPa), 10 Hz (574,0 MPa) e 20 Hz (605,1 MPa). Portanto, o uso de frequências de até 20 Hz pode ser tido como uma alternativa para acelerar ensaios de resistência à fadiga em cerâmicas policristalinas.

Palavras-chave: Cerâmicas. Fadiga. Frequência. Resistência. Usinagem.

ABSTRACT

FATIGUE BEHAVIOR OF CERAMICS

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Two manuscripts about the fatigue behavior of ceramic materials are reported in this thesis. Manuscript 1: Impact of the CAD/CAM machining on the fatigue behavior of glass and polycrystalline ceramics. This study assessed the effect of CAD/CAM machining on the flexural fatigue strength and on the surface roughness of different ceramics by comparing machined vs. machined and polished specimens. Disc shaped specimens of Y-TZP, leucite- and lithium disilicate-based glass ceramics were prepared by CAD/CAM machining, and assigned into two groups: machining (M); and machining followed by polishing (MP). The surface roughness (Ra and Rz) was measured in a contact perfilometer and the flexural fatigue strength was evaluated by the step-test method (n = 20), using a piston-on-three ball assembly (ISO 6872:2008). A specific loading protocol, based on the results of a monotonic test (n = 5), was performed for each ceramic material. A maximum of 10,000 cycles was applied in each load step, at 1.4 Hz. Weibull probability statistics was used for the analysis of the fatigue flexural strength, and the non-parametric Mann-Whitney test ($\alpha = 0.05$) was used to compare the roughness values (Ra and Rz) between M and MP groups for each ceramic material. M resulted in higher values of roughness and lower values of characteristic fatigue strength than MP. The greatest reduction in the characteristic fatigue strength from MP to M was observed in Y-TZP (40%; M = 536.48 MPa; MP = 894.50 MPa), followed by lithium disilicate (33%; M = 187.71 MPa; MP = 278.93 MPa) and leucite glass ceramic (29%; M = 72.61 MPa; MP = 102.55 MPa). Therefore, CAD/CAM machining affected negatively the flexural fatigue strength of ceramics with different microstructures. The results suggest that the machining of partially sintered materials may be as deleterious as the machining of fully sintered materials. Manuscript 2: Loading frequencies up to 20 Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic. This study aimed to investigate the influence of the loading frequency on the zirconia fatigue strength, by means of the staircase approach, at a maximum lifetime of 500.000 cycles, and using a piston-on-three ball assembly (ISO 6872:2008). The frequencies investigated were 2 Hz (control - simulation of the chewing activity; n = 20), 10 Hz (n = 20), 20 Hz (n = 20), and 40 Hz (n = 21). The fatigue strength data were analyzed using one-way ANOVA and post hoc Tukey`s test ($\alpha = 0.05$). The fatigue strength was significantly higher for 40 Hz group (630.7 MPa) and did not differ among the groups 2 Hz (550.3 MPa), 10 Hz (574.0 MPa) and 20 Hz (605.1 MPa). Therefore, the use of loading frequencies up to 20 Hz seems a good alternative to expedite the cycling strength fatigue tests in polycrystalline ceramics.

Keywords: Ceramics. Fatigue. Frequency. Machining. Strength.

SUMÁRIO

1 INTRODUÇÃO

As cerâmicas odontológicas são reconhecidas pela biocompatibilidade, durabilidade química e excelentes propriedades ópticas, sendo capazes de mimetizar adequadamente a estrutura dental. Em função disso, esses materiais têm ganhado papel de destaque na Odontologia Restauradora como forma de satisfazer a crescente exigência estética por parte dos pacientes (KELLY, 2004).

O desenvolvimento de cerâmicas com melhores propriedades mecânicas, aliado a implementação da tecnologia CAD/CAM (*Computer-Aided Design/Computer-Aided Machining*), viabilizou a confecção de trabalhos protéticos totalmente cerâmicos, os quais variam desde restaurações unitárias, como facetas, *inlays*, *onlays* e coroas, até complexas próteses parciais fixas envolvendo múltiplos elementos (KELLY; NISHIMURA; CAMPBELL, 1996).

Nos sistemas CAD/CAM, a usinagem de restaurações cerâmicas pode ser realizada a partir de blocos pré-fabricados densamente sinterizados, sendo denominada usinagem de corte duro, ou utilizando blocos parcialmente sinterizados, corte macio. As cerâmicas comumente disponíveis para usinagem de corte duro são feldspática, vítrea reforçada por leucita e dissilicato de lítio. Novas formulações para usinagem de corte duro têm sido introduzidas no mercado, a exemplo da cerâmica vítrea de silicato de lítio reforçada por óxido de zircônio. Cerâmicas de maior dureza, como a zircônia parcialmente estabilizada por óxido de ítrio (Y-TZP), são preferencialmente usinadas antes da completa sinterização, a fim de facilitar o processo de fresagem (DENRY; HOLLOWAY, 2010; DENRY; KELLY, 2014).

Apesar do desenvolvimento de materiais com maior resistência e tenacidade à fratura, como a zircônia e a alumina policristalina, a natureza frágil e o crescimento lento e subcrítico de trincas são características inerentes às cerâmicas, fazendo com que a resistência à fratura desses materiais seja fortemente dependente do tamanho, número e distribuição de defeitos (HONDRUM, 1992; KELLY, 1995).

As taxas de sucesso clínico reportadas para restaurações totalmente cerâmicas são promissoras, muito embora a fratura da cerâmica ainda seja um problema técnico recorrente. Recente revisão sistemática, com 67 estudos clínicos incluídos, reportou que a taxa de fratura da infraestrutura foi significativamente superior para coroas totalmente cerâmicas em comparação às coroas metalocerâmicas, para uma estimativa de cinco anos. Nas cerâmicas de menor resistência, como as feldspáticas, a taxa de fratura da infraestrutura foi de 6,7% (IC[1] 95%: 2,4% - 17,7%). Nas cerâmicas vítreas reforçadas por leucita e dissilicato de lítio, essa taxa foi de 2,3% (IC 95%: 1,0% - 5,5%), enquanto que nas cerâmicas de maior resistência, como a zircônia, essa taxa foi de apenas 0,4% (IC 95%: 0,1% - 1,7%), resultado estatisticamente similar ao obtido para coroas metalocerâmicas (0,03%; IC 95%: 0,002% - 0,3%) (SAILER et al., 2015).

A análise fractográfica de coroas que falharam clinicamente e a análise de elementos finitos indicam que a superfície de cimentação da peça cerâmica concentra grande parte das tensões de tração responsáveis pelo início da falha em coroas cerâmicas (KELLY et al., 1990; THOMPSON et al., 1994; QUINN et al., 2005; MAY et al., 2012). Desse modo, torna-se imperativo a avaliação do efeito de procedimentos que afetam diretamente regiões de concentração de tensão de tração na resistência à fratura das cerâmicas.

A redução de blocos cerâmicos, por instrumentos diamantados de corte, na anatomia da restauração desejada, é capaz de introduzir microtrincas e defeitos na superfície da cerâmica (FRAGA et al., 2015), com dimensão estimada em 9 a 15 µm para uma cerâmica vítrea reforçada por partículas (KELLY et al., 1991), e em até 60 µm para uma cerâmica feldspática (SINDEL et al., 1998). Esses defeitos serão pontos de concentração de tensões, podendo dar origem a fratura da restauração. Dessa forma, considerando-se o uso cada vez mais difundido dos sistemas CAD/CAM na Odontologia Restauradora e o potencial da usinagem de introduzir defeitos em regiões críticas de concentração de tensão de tração, a exemplo da superfície de cimentação de coroas, o estudo do efeito da usinagem CAD/CAM na resistência à fratura de cerâmicas com diferentes microestruturas faz-se necessário.

Tendo-se em vista os constantes desafios a que os materiais restauradores estão sujeitos no meio bucal, como presença de umidade, cargas mastigatórias e alterações de temperatura e pH, as restaurações tendem a falhar por um fenômeno chamado fadiga (WISKOTT; NICHOLLS; BELSER, 1995), o qual compreende a falha de um material sujeito a tensões ou deformações ao longo de um período de tempo (BARAN; BOBERICK; McCOOL, 2001). No que se refere ao estudo das propriedades mecânicas dos materiais dentários, os ensaios de fadiga cíclica se colocam como um complemento aos ensaios monotônicos tradicionais, reproduzindo uma condição mais próxima do que ocorre clinicamente (WISKOTT; NICHOLLS; BELSER, 1995).

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[1] IC: Intervalo de Confiança.

A realização de ensaios de fadiga cíclica envolve a determinação prévia de algumas variáveis, a exemplo da frequência de aplicação da carga (número de ciclos realizados por segundo), cujo efeito na resistência à fadiga de materiais cerâmicos ainda não é totalmente compreendido (ROSENTRITT et al., 2006; KELLY et al., 2010; JOSHI et al., 2014).

Segundo Wiskott, Nicholls e Belser (1995), para apresentar relevância clínica, os ensaios de fadiga deveriam ser conduzidos com, no mínimo, 10^6 ciclos, o que simularia um ano em boca. Empregando-se uma frequência de 2 Hz (frequência do ciclo mastigatório varia de 0,94 Hz a 2,15 Hz, segundo Po et al. (2011)), seriam necessários cerca de 6 dias para concluir a ciclagem em apenas um corpo de prova. Se uma frequência de 20 Hz pudesse ser utilizada, sem prejuízos aos resultados, esse tempo se reduziria para 14 horas. A aceleração dos ensaios cíclicos viabilizaria a aplicação de um número total de ciclos com maior relevância clínica, bem como a utilização de mais espécimes, contribuindo para aumentar o grau de evidência dos estudos de fadiga dos materiais cerâmicos.

Tendo-se em vista a existência de literatura escassa, o grande tempo dispendido para a realização de ensaios cíclicos com frequências baixas e o fato de muitos trabalhos na área de materiais dentários utilizarem critérios pouco elucidativos para a determinação da frequência, aponta-se para a necessidade de se avaliar a influência da aceleração do ensaio cíclico, por meio do aumento da frequência de aplicação de carga, sobre a resistência à fadiga das cerâmicas odontológicas.

Assim, considerando-se os contextos expostos, no presente trabalho serão apresentados os artigos oriundos de duas investigações científicas. O primeiro deles, intitulado "***Impact of the CAD/CAM machining on the fatigue behavior of glass and polycrystalline ceramics***", visou avaliar o efeito da usinagem sobre a resistência à fadiga flexural de cerâmicas com diferentes microestruturas (vítrea reforçada por leucita, dissilicato de lítio e Y-TZP), a partir da comparação entre corpos de prova usinados por CAD/CAM e corpos de prova com superfície polida após usinagem. O segundo artigo, intitulado "***Loading frequencies up to 20 Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic***" investigou o efeito da frequência de aplicação de carga (número de ciclos por segundo) na resistência à fadiga flexural da cerâmica policristalina Y-TZP e objetivou determinar a frequência máxima para aplicação de carga que não alterasse significativamente a resistência à fadiga flexural apresentada pelas cerâmicas na frequência de 2 Hz (simulação aproximada da frequência do ciclo mastigatório).

2 REVISÃO DE LITERATURA

2.1 AS CERÂMICAS COMO MATERIAIS ODONTOLÓGICOS RESTAURADORES

As cerâmicas são formadas por elementos metálicos e não-metálicos. No estado líquido, esses elementos movem-se livremente, enquanto que, no processo de solidificação, eles tendem a se arranjar em uma estrutura vítrea amorfa ou em uma estrutura cristalina organizada (LAWSON; BURGESS, 2014). As cerâmicas odontológicas podem ser definidas como composições de duas ou mais fases, em que uma matriz vítrea é reforçada por partículas cristalinas ou vítreas de alta fusão. Nas cerâmicas sem conteúdo vítreo, chamadas policristalinas, a matriz é constituída por óxidos de alumínio ou zircônio, sendo acrescidos átomos modificadores ou estabilizadores de fase (KELLY, 2008).

De acordo com a microestrutura, as cerâmicas podem ser classificadas como: predominantemente vítreas, vítreas reforçadas por partículas e policristalinas. As cerâmicas predominantemente vítreas são compostas por uma matriz a base de vidro, a exemplo do feldspato, na qual são impregnadas pequenas quantidades de partículas que melhoram suas propriedades térmicas e ópticas. A presença de um alto conteúdo vítreo, apesar de diminuir sua resistência à fratura, proporciona excelente estética e capacidade de mimetizar os tecidos dentais. As cerâmicas vítreas reforçadas por partículas são aquelas em que componentes cristalinos ou vítreos de alta fusão são adicionados a sua composição visando melhorar propriedades mecânicas, como resistência e expansão/contração térmicas. Esses componentes podem ser adicionados mecanicamente ou serem precipitados dentro da matriz vítrea por meio de tratamento térmico. Como exemplos de partículas, pode-se citar: leucita, óxido de alumínio, óxido de zircônio e dissilicato de lítio. As cerâmicas policristalinas não apresentam matriz vítrea e seus componentes encontram-se densamente arranjados em uma estrutura cristalina, o que lhes confere maior tenacidade à fratura e resistência. Em contrapartida, apresentam propriedades ópticas inferiores, sendo mais opacas. Como principais exemplos desses materiais podem-se citar a alumina policristalina e a zircônia parcialmente estabilizada por óxido de ítrio (Y-TZP) (KELLY, 2004, 2008; LAWSON; BURGESS, 2014).

Nas investigações científicas descritas nesse trabalho foram utilizados representantes de dois tipos de microestruturas de cerâmicas, disponíveis na forma de blocos pré-fabricados para usinagem pelo sistema CAD/CAM, a saber: cerâmica vítrea reforçada por leucita, cerâmica vítrea reforçada por dissilicato de lítio e zircônia parcialmente estabilizada por óxido de ítrio.

A cerâmica vítrea reforçada por leucita para CAD/CAM (IPS Empress CAD, Ivoclar Vivadent, Liechtenstein) é indicada pelo fabricante para confecção de facetas, *inlays*, *onlays* e coroas. Essa cerâmica caracteriza-se por uma matriz vítrea de aluminosilicato, na qual ficam impregnados cristais de leucita ($K_2O.Al_2O_3.4SiO_2$), com diâmetros de 1 a 5 µm, totalizando 35-45% de volume da fase cristalina. Esses cristais são adicionados no intuito de melhorar as propriedades mecânicas do material, uma vez que atuam nos mecanismos de tenacificação de fratura, por promoverem o desvio da trajetória de propagação da trinca (CESAR et al., 2005). A cerâmica vítrea reforçada por leucita para CAD/CAM é comercialmente disponível na forma de blocos totalmente sinterizados (IVOCLAR VIVADENT, 2006a).

A cerâmica de dissilicato de lítio para CAD/CAM (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) alia excelentes propriedades ópticas e resistência à fratura superior à cerâmica vítrea reforçada por leucita. É indicada pelo fabricante para a confecção de facetas, *inlays*, *onlays*, coroas e intermediários de implantes para restaurações unitárias (IVOCLAR VIVADENT, 2006b). Os blocos são disponibilizados na forma pré-cristalizada, conhecida como *blue stage*, a qual contém metassilicato e núcleos de dissilicato de lítio. Nessa fase, os blocos são mais facilmente usinados e, após a confecção, a restauração é submetida a um tratamento térmico, ocorrendo a completa cristalização do dissilicato de lítio (DENRY, 2013; LI; CHOW; MATINLINNA, 2014).

Uma das cerâmicas de maior resistência à fratura é a zircônia parcialmente estabilizada por óxido de ítrio (Y-TZP), cujo uso em Odontologia foi possível graças a implementação dos sistemas CAD/CAM (LI; CHOW; MATINLINNA, 2014). A zircônia pode assumir três formas cristalográficas, a depender da temperatura: monoclínica (da temperatura ambiente à 1170ºC), tetragonal (entre 1170ºC e 2370ºC) e cúbica (acima de 2370ºC). Durante o resfriamento, a passagem da fase tetragonal para a monoclínica é acompanhada por um considerável aumento no volume (aproximadamente 4,5%), o qual é suficiente para ocasionar a falha catastrófica do material. A adição de óxidos como CaO, MgO, Y_2O_3 ou CeO_2 tornou possível estabilizar a fase tetragonal a temperatura ambiente, permitindo o uso da zircônia como material estrutural (DENRY; KELLY, 2008; LI; CHOW; MATINLINNA, 2014). A zircônia estabilizada exibe um mecanismo de tenacificação da fratura por mudança de fase induzida por tensão. Quando uma tensão se concentra em torno de uma trinca, há uma mudança de fase tetragonal para monoclínica dos grãos na ponta da trinca. Essa mudança é seguida por aumento no volume dos grãos, induzindo tensões compressivas e dificultando a propagação da trinca (CHEVALIER; GREMILLARD; DEVILLE, 2007). A Y-TZP é amplamente empregada na confecção de coroas e próteses parciais fixas (DENRY; KELLY, 2008). Pode ser usada como infraestrutura, sendo recoberta com cerâmica vítrea, ou como restauração monolítica (DENRY; KELLY, 2008; TONG et al., 2016).

2.2 PROPRIEDADES MECÂNICAS DAS CERÂMICAS ODONTOLÓGICAS

As cerâmicas caracterizam-se por serem materiais frágeis, ou seja, suportam pouca ou nenhuma deformação plástica antes de fraturar. Em função disso, são sensíveis a presença de defeitos, apresentam menor resistência à tração quando comparada à compressão e fraturam de maneira repentina (catastrófica) (HONDRUM, 1992).

Os defeitos presentes no material poderão atuar como pontos de concentração de tensões, dando início a propagação de uma trinca quando a energia elástica armazenada exceder a energia requerida para a formação de uma nova superfície. Dessa forma, a resistência das cerâmicas depende do tamanho, número e distribuição de defeitos na zona de maior concentração de tensão de tração, bem como da capacidade do material de resistir à propagação rápida da trinca (tenacidade à fratura) (KELLY, 1995). Exemplos de defeitos que podem ser encontrados nas cerâmicas são: poros, trincas, inclusões, além de defeitos superficiais decorrentes de desgaste e usinagem (QUINN, 2007; QUINN; HOFFMAN; QUINN, 2012; DENRY, 2013).

Outras variáveis também podem influenciar nos valores de resistência das cerâmicas, como: espessura da camada de cimento (MAY et al., 2012), condições de carregamento do ensaio, ambiente do teste (seco; úmido) geometria e volume do corpo de prova (KELLY, 1995). Em função disso, a resistência à fratura dos materiais cerâmicos é tida como uma medida condicional.

2.3 COMPORTAMENTO CLÍNICO DAS RESTAURAÇÕES TOTALMENTE CERÂMICAS

Informações sobre cerca de 35.000 restaurações totalmente cerâmicas confeccionadas em sistema CAD/CAM foram coletadas de um banco de dados pertencente a um centro de usinagem na Alemanha. Devido ao fato das peças usinadas apresentarem garantia de cinco anos, a ocorrência de fratura era reportada pelos dentistas e registrada na base de dados do centro. Os tipos de restauração compreendiam *inlays*, *onlays*, coroas unitárias e próteses parciais fixas (de 3 a 5 elementos) cimentadas apenas em dentes posteriores. O sistema cerâmico empregado variou de acordo com o tipo de trabalho restaurador. Zircônia monolítica e infraestrutura de zircônia recoberta por dissilicato de lítio (*sistema e.max CAD on* ZrO_2) foram utilizadas na confecção de próteses parciais fixas e coroas unitárias, enquanto que zircônia recoberta com cerâmica feldspática foi utilizada apenas para a confecção de próteses parciais fixas. Dissilicato de lítio foi empregado na confecção de coroas, *onlays* e *inlays*, enquanto que cerâmica vítrea reforçada por leucita apenas foi utilizada para confecção de *onlays* e *inlays*. No período de 3,5 anos, 491 restaurações totalmente cerâmicas fraturaram (1,4%). Em relação aos tipos de cerâmica, para o período observado, o dissilicato de lítio apresentou taxa de sobrevivência inferior para as coroas unitárias (1,23% de falha) em comparação às *onlays* (0,71% de falha) e *inlays* (0,36% de falha). Em relação às próteses parciais fixas, não houve diferença na análise de sobrevivência entre a zircônia recoberta por disilicato de lítio (3,93% de falha), a zircônia recoberta por porcelana (0,82% de falha) e a zircônia monolítica (nenhuma falha). Cerâmica vítrea reforçada por leucita apresentou taxa de sobrevivência inferior para restaurações tipo *inlays* (3,08% de falha) e *onlays* (3,57% de falha) quando comparada ao dissilicato de lítio (BELLI et al., 2016).

Em recente revisão sistemática, na qual 67 estudos clínicos foram incluídos, a taxa de sobrevivência para coroas unitárias metalocerâmicas foi estimada em 94,7% (IC 95%: 94,1% - 96,9%) para um período de cinco anos. Essa taxa de sobrevivência foi similar às reportadas para coroas unitárias confeccionadas com cerâmicas vítreas reforçadas por leucita ou dissilicato de lítio (96,6%; IC 95%: 94,9% - 96,7%), alumina infiltrada por vidro (94,6%; IC 95%: 92,7% - 96,0%), alumina densamente sinterizada (96,0; IC 95%: 93,8% - 97,5%) e zircônia (91,2%, IC 95%: 82,8% - 95,6%). Entretanto, taxas de sobrevivência significativamente inferiores foram relatadas para coroas unitárias confeccionadas com cerâmicas a base de feldspato e sílica (90,7%; IC 95%: 87,5% - 93,1%). Salienta-se que, no estudo mencionado, sobrevivência foi definida como restaurações que permaneciam em boca com ou sem modificações durante o período de observação. Fratura catastrófica foi uma das complicações técnicas mais reportadas para as coroas totalmente cerâmicas. A taxa de fratura para cinco anos foi estimada em 6,7% para as cerâmicas feldspáticas, 2,3% para as cerâmicas vítreas reforçadas por leucita e dissilicato de lítio, e 0,4% para as cerâmicas policristalinas (SAILER et al. 2015).

No que se refere a próteses parciais fixas (PPFs), revisão sistemática com 40 estudos incluídos, mostrou uma taxa de fratura da cerâmica de infraestrutura superior para cerâmicas vítreas reforçadas por dissilicato de lítio (8%) e alumina infiltrada por vidro (12,9%), quando comparada a PPFs metalocerâmicas (0,6%) e de zircônia (1,9%) (PJETURSSON et al., 2015).

2.4 ANÁLISE FRACTOGRÁFICA DE RESTAURAÇÕES CERÂMICAS QUE FALHARAM CLINICAMENTE

Alguns trabalhos têm reportado a análise fractográfica de restaurações totalmente cerâmicas que falharam clinicamente, fornecendo informações importantes quanto ao modo de falha e à origem do defeito crítico. Essa análise mostra-se desafiadora, uma vez que muitos fatores podem estar associados com o início e a propagação de trincas em restaurações cerâmicas, incluindo: forma da restauração, heterogeneidades microestruturais, tamanho e distribuição de falhas superficiais, tensão residual e gradientes de tensão induzidos por polimento e/ou processamento térmico, espessura da cerâmica, módulo de elasticidade dos componentes da restauração, interface cimento/cerâmica, magnitude e orientação das cargas aplicadas (THOMPSON et al., 1994).

Kelly et al. (1990) avaliaram 12 coroas de cerâmica vítrea (Dicor, Dentsply International Inc., York, PA) e de espinélio de alumínio e magnésio (Cerestore, Ceramco Inc., East Windsor, NJ) que falharam clinicamente na consulta de prova ou após o período de 17 a 36 meses de uso. Na maioria das coroas, a falha aparentemente teve início a partir da superfície interna, indicando uma alta concentração de tensão de tração nessa região associada à presença de defeitos críticos. As falhas foram relacionadas com defeitos incorporados durante a fabricação ou a defeitos inerentes a microestrutura do material.

Thompson et al. (1994) encontraram resultados similares para nove coroas de cerâmica vítrea (Dicor, Dentsply International Inc., York, PA) e nove coroas de espinélio de alumínio e magnésio (Cerestore, Ceramco Inc., East Windsor, NJ) que falharam clinicamente no período de seis a 48 meses após cimentação com fosfato de zinco. Todas as coroas Dicor® falharam a partir de algum defeito na superfície interna. Os autores forneceram duas explicações para esse tipo de comportamento: 1) o jateamento com partículas abrasivas de alumina, antes da cimentação, poderia ter originado defeitos que atuaram como concentradores de tensões, facilitando o início da falha nesse local; 2) a falha do cimento na interface restauração/cimento poderia aumentar a flexão dentro da coroa durante carregamento na superfície oclusal, ocasionando tensão de tração suficiente para induzir o início da falha na superfície interna. Em sete das nove coroas de Cerestore®, a falha teve início na interface cerâmica de cobertura/cerâmica de infraestrutura ou dentro da cerâmica de infraestrutura.

Quinn et al. (2005) realizaram a análise fractográfica de três coroas totalmente cerâmicas que falharam clinicamente, compostas por cerâmica de infraestrutura e de cobertura. Na coroa correspondente ao primeiro molar superior direito, constatou-se que a falha teve início na cerâmica de infraestrutura de alumina densamente sinterizada (Procera AllCeram, Nobel Biocare, Suécia), próximo às margens da restauração, onde o material é mais fino e há concentração de tensões. Mecanismo de falha semelhante foi observado na coroa de espinélio de alumínio e magnésio (Cerestore, Coors Biomedical, Lakewood, CO), em primeiro molar inferior esquerdo, onde a fratura pareceu iniciar na cerâmica de infraestrutura, na vizinhança da área marginal. Ambas as coroas continham *cone cracks* (trincas que resultam do contato brusco de forças aplicadas em superfícies relativamente planas) na superfície oclusal. Na coroa de incisivo central superior direito de dissilicato de lítio injetável (Empress 2, Ivoclar Vivadent, Liechtenstein), *wake hackles* partindo diretamente da infraestrutura em direção a superfície indicavam que a falha teve início na cerâmica de infraestrutura, na face lingual do dente.

Øilo e Gjerdet (2013) analisaram a superfície de fratura de 27 coroas de alumina revestida por cerâmica vítrea que falharam clinicamente, compreendendo 13 incisivos, 3 pré-molares e 11 molares. Em todos os casos, a falha iniciou na cerâmica de infraestrutura na margem cervical da coroa. Muitas das restaurações apresentavam fratura por dano de contato na face oclusal da cerâmica de cobertura. Entretanto, todas essas falhas foram consideradas secundárias, não sendo responsáveis pela falha catastrófica das coroas. Em recente estudo (ØILO; QUINN, 2016), essas coroas foram re-avaliadas e a origem da fratura identificada. A maioria das falhas teve início a partir de defeitos localizados nas margens das restaurações usinadas por CAD/CAM, as quais se mostravam muito finas, irregulares e com excesso de cerâmica de cobertura na porção interna. Em alguns casos, as marcas da usinagem eram bem evidentes e correspondiam a trincas e lascamentos responsáveis pelo início da falha.

2.5 EMPREGO DA TECNOLOGIA CAD/CAM NA CONFECÇÃO DE RESTAURAÇÕES CERÂMICAS

A tecnologia CAD/CAM se coloca como uma alternativa aos métodos tradicionais de confecção de restaurações cerâmicas, os quais tendem a ser mais demorados e dependentes da habilidade manual do técnico em prótese dentária (MIYAZAKI et al., 2009). Os sistemas CAD/CAM empregam blocos cerâmicos pré-fabricados, os quais são obtidos por um processo controlado e padronizado, resultando em uma cerâmica mais homogênea e com menores chances de incorporação de defeitos (GIORDANO, 2006; LI; CHOW; MATINLINNA, 2014).

De forma geral, a confecção de uma restauração cerâmica completa ou de sua infraestrutura por tecnologia CAD/CAM abrange três momentos: (1) digitalização do preparo, por meio do uso de câmeras intraorais ou escaneamento do modelo de trabalho; (2) desenho digital da restauração e (3) usinagem da peça a partir do desgaste de um bloco cerâmico com instrumentos diamantados de corte (BEUER; SCHWEIGER; EDELHOFF, 2008).

Vários sistemas CAD/CAM são comercializados para a usinagem das cerâmicas odontológicas, sendo o CEREC (Sirona Dental Systems Gmbh, Alemanha) um dos mais amplamente difundidos e estudados (LEBON et al., 2016). Inicialmente, esse sistema permitia apenas a confecção de restaurações do tipo *inlay* por meio do desgaste de blocos cerâmicos por discos diamantados. Atualmente, utiliza pontas diamantadas específicas para a usinagem de blocos de resina composta, materiais híbridos, cerâmicas vítreas e blocos parcialmente sinterizados de Y-TZP, permitindo a confecção dos mais variados tipos de restaurações indiretas como *inlays*, *onlays*, coroas e próteses parciais fixas (MÖRMANN, 2006).

2.5.1 Impacto da usinagem na resistência à fratura e na rugosidade superficial de cerâmicas CAD/CAM

A usinagem pode ser realizada a partir de blocos cerâmicos totalmente sinterizados (corte duro), ou utilizando-se blocos parcialmente sinterizados (corte macio). Cerâmicas feldspática, vítrea reforçada por leucita, dissilicato de lítio e cerâmicas vítreas de silicato de lítio reforçadas por óxido de zircônio são alguns dos materiais disponíveis para confecção de restaurações a partir de fresagem de corte duro (DENRY, 2013; DENRY; KELLY, 2014). Já a zircônia tetragonal parcialmente estabilizada com óxido de ítrio pode ser usinada por fresagem de corte macio, a partir de blocos parcialmente sinterizados, ou por meio de fresagem de corte duro, utilizando-se blocos densamente sinterizados (DENRY, 2013; DENRY; KELLY, 2014).

De uma forma geral, a usinagem da cerâmica por instrumentos diamantados de corte compreende cinco estágios. Primeiramente tem-se a indução de um campo de concentração de tensões na cerâmica, em função do impacto gerado pelo primeiro contato entre o instrumento de corte e a cerâmica. Em um segundo momento, a movimentação do instrumento provoca o acúmulo de energia e o início da formação de trincas nas regiões de alta concentração de tensão. O terceiro estágio compreende a propagação das trincas em função do progresso da usinagem. O processo de propagação de trincas é completado em um quarto estágio, quando múltiplas microtrincas se fundem e promovem a remoção do material cerâmico, culminando com o alívio da energia armazenada. Por fim, ter-se-á, em um quinto momento, a formação de uma nova textura de superfície na cerâmica usinada, a qual contará com a presença de danos superficiais e sub-superficiais (ZHANG; SATISH; KO, 1994), cuja extensão foi estimada em até 60 µm para uma cerâmica feldspática usinada pelo sistema CEREC1® (SINDEL et al., 1998).

Fraga et al. (2015) mostraram que a usinagem de corte duro reduziu em cerca de 27% a resistência flexural biaxial de uma cerâmica vítrea reforçada por leucita (128,20 MPa), quando comparada à usinagem seguida de polimento (177,2 MPa). Por meio de imagens em microscopia eletrônica de varredura (MEV) da secção transversal dos discos, observou-se que a usinagem foi capaz de introduzir defeitos na superfície da cerâmica, os quais foram removidos com a realização do polimento. A rugosidade média (Ra) do grupo usinado foi de 1,37 µm, enquanto do grupo usinado e polido foi de 0,04 µm.

A análise fractográfica de barras de cerâmica feldspática e vítrea reforçada por partículas, submetidas a ensaio de flexão três pontos, mostraram que todas as 72 barras analisadas 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 por partículas e 15-30 µm para a cerâmica feldspática (KELLY et al.,1991).

Em ensaio de flexão uniaxial, barras de Y-TZP obtidas por usinagem pelo sistema Cercon (Degudent GmbH, Alemanha) apresentaram resistência característica de 820,65 MPa e o módulo de Weibull de 3,99, valores inferiores ao grupo que recebeu apenas polimento, os quais mostraram uma resistência característica de 1244,17 MPa e módulo de Weibull de 7,26. O valor médio de Ra após usinagem foi de 1,91 µm, enquanto para os corpos de prova polidos foi de 0,04 µm (WANG; ABOUSHELIB; FEILZER, 2008).

Tem sido relatado na literatura que a usinagem poderia gerar uma fina faixa de tensão compressiva sobre a superfície da cerâmica, dificultando a propagação de trincas e promovendo melhoria na resistência do material (MARSHALL et al., 1983).

No que se refere ao impacto de variáveis relacionadas à usinagem sobre as propriedades mecânicas dos materiais cerâmicos, nos trabalhos de Addison et al. (2012) e Fraga et al. (2015) observou-se que a rugosidade superficial e a resistência flexural de discos obtidos por usinagem de corte duro em sistema CEREC MC XL® foi influenciada pelo par de brocas usado. Mesmo sendo idênticas em composição e geometria, diferenças na distribuição e orientação das partículas de diamante impregnadas na superfície das brocas podem explicar tais resultados. Fraga et al. (2015) relataram moderada e significativa correlação entre ordem de usinagem e rugosidade, sendo que, à medida que a ordem de usinagem aumentou, os valores de rugosidade tenderam a diminuir. Tais achados foram contrários ao estudo de Addison et al. (2012), onde não se encontrou correlação. Esta contradição entre os resultados, pode ser devido ao menor número de discos produzidos por broca no trabalho de Adisson et al. (2012) (n=14) em comparação ao de Fraga et al. (2015) (n=28).

Corazza et al. (2015) investigaram a influência da deterioração das brocas CAD/CAM na rugosidade e na carga máxima para falha de infraestruturas de Y-TZP recobertas com porcelana feldspática. A rugosidade dos corpos de prova variou entre os dois pares de brocas usados na fresagem. Entretanto, essa diferença não se refletiu nos valores de carga para fratura. Apesar de significativa, a correlação entre ordem de usinagem e rugosidade foi moderada.

Salienta-se que, apesar da expansão na utilização de sistemas CAD/CAM, não foram encontrados estudos de fadiga que tenham avaliado o efeito a longo prazo da usinagem na resistência à fratura das cerâmicas. Portanto, este efeito permanece desconhecido.

2.6 FADIGA EM CERÂMICAS

O termo fadiga designa um processo progressivo de dano ocorrido em um material sujeito a tensões ou deformações ao longo de um período de tempo, resultando na propagação de trincas e na falha do material (BONFANTE; COELHO, 2016). O modo de aplicação da tensão ou deformação pode ser: estático, dinâmico ou cíclico (BARAN; BOBERICK; McCOOL, 2001). A fadiga estática refere-se ao crescimento de trincas sob a aplicação de uma tensão estática constante em um ambiente químico de fragilização (SURESH, 1998). Nos ensaios de fadiga dinâmica, a susceptibilidade do material ao crescimento subcrítico de trincas é determinada por meio de testes de resistência em que a tensão é aplicada interruptamente, sob uma taxa constante (exemplo: 10^{-2}, 10^{-1}, 10^{0}, 10^{1} e 10^{2} MPa/s) (THOMPSON, 2004). Já a fadiga cíclica refere-se a perda de resistência do material sob carregamento cíclico, podendo levar a falhas sob tensões inferiores a resistência nominal do material (BONFANTE; COELHO, 2016).

Em função de sua natureza frágil, a falha por fadiga das cerâmicas ocorre de maneira súbita (catastrófica), sem ser precedida de qualquer deformação plástica visível, abrangendo três estágios distintos: 1) nucleação da trinca; 2) propagação; 3) coalescência (CALLISTER, 1991; BARAN; BOBERICK; McCOOL, 2001).

O primeiro estágio, nucleação da trinca, refere-se à formação da trinca a partir de um ponto de alta concentração de tensão, como poros, danos superficiais, ângulos agudos, inclusões e agregados (QUINN, 2007). O processo de nucleação é seguido por um rápido crescimento da trinca. No segundo estágio, a trinca tende a se propagar com velocidade inferior e proporcional a tensão recebida, sendo a propagação perpendicular ao eixo de tração (BARAN; BOBERICK; McCOOL, 2001). O estágio três de coalescência caracteriza-se pela falha final, que ocorre muito rapidamente após a trinca ter atingido um tamanho crítico (CALLISTER, 1991).

Os materiais cerâmicos são suscetíveis ao fenômeno de crescimento lento e subcrítico de trincas (*Slow Crack Growth* – SCG). O SCG ocorre por uma interação química entre a cerâmica e o ambiente, na presença de umidade e tensões, em que a trinca se propaga de modo estável a partir de um valor de intensidade de tensão abaixo do nível crítico (K_{I0}), culminando na diminuição da resistência do material em função do tempo (GONZAGA et al., 2011).

Diante dos constantes desafios a que os materiais restauradores estão sujeitos no ambiente oral, a exemplo de presença de umidade, forças mastigatórias, forças imprimidas pelos lábios, língua e bochechas, além de variações de temperatura e pH, torna-se fácil compreender o porquê de a fadiga ser a principal causa de falhas clínicas de trabalhos protéticos (WISKOTT; NICHOLLS; BELSER, 1995). Desse modo, a compreensão de como esse fenômeno ocorre é de fundamental importância para que se possa fazer inferências sobre a probabilidade de falha dos materiais restauradores (LOHBAUER et al., 2008).

2.6.1 Ensaios de fadiga

Tendo-se em vista os altos custos, as considerações éticas, e o tempo dispendido na realização de trabalhos clínicos (KELLY, 2016), nos últimos anos, tem-se observado um interesse crescente por parte da comunidade científica no estudo do comportamento à fadiga dos materiais cerâmicos (ROSENTRITT; BEHR; PREIS, 2016; BONFANTE; COELHO, 2016; BARAN; BOBERICK; McCOOL, 2001). Os ensaios de fadiga têm se colocado como uma alternativa para aproximar os testes laboratoriais da realidade clínica a que os materiais restauradores são submetidos, sendo utilizados para a comparação entre materiais e realização de análises de sobrevivência.

O comportamento à fadiga dos materiais cerâmicos pode ser avaliado por meio de diferentes metodologias, sendo que a escolha do tipo de ensaio recai na informação que se deseja obter. Cargas mecânicas cíclicas 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 (ROSENTRITT; BEHR; PREIS, 2016). Cargas estáticas ou dinâmicas podem ser vistas em metodologias que visam informações acerca do comportamento de crescimento de trincas dos materiais cerâmicos (GONZAGA et al., 2011; THOMPSON, 2004).

De acordo com Collins (1993), o termo resistência à fadiga compreende o valor de tensão em que a falha ocorrerá após um período pré-determinado. Já o termo limite de fadiga compreende o valor de tensão abaixo do qual o material resistiria a um número infinito de ciclos.

Alguns dos métodos que têm sido usados para a investigação da resistência à fadiga cíclica das cerâmicas são o *staircase* e o *step-test*.

No método *staircase*, também conhecido como *up and down approach*, as amostras são testadas sequencialmente para determinação da média e do desvio padrão da resistência à fadiga. Nessa abordagem, o número máximo de ciclos é previamente estabelecido, tendo-se por base a experiência do pesquisador e uma estimativa do número de ciclos a que o componente será sujeito ao longo de seu uso. A primeira amostra é testada com valor de tensão inferior a resistência nominal máxima do material em teste estático correspondente. Se a amostra falhar, o próximo espécime é testado com incremento de carga inferior; se a amostra sobreviver, o próximo espécime é testado com incremento de carga superior. Portanto, cada teste é dependente dos resultados do teste anterior. Como o ensaio é sequencial, tende a ser centrado no valor médio de resistência à fadiga para o número de ciclos determinado, o qual será calculado com base no evento menos frequente (falha ou sobrevivência). No mínimo 15 corpos de prova devem ser testados após o início do ensaio, sendo este representado pela ocorrência da primeira inversão de eventos (fratura seguida de sobrevivência, ou vice-versa) (COLLINS, 1993).

O método *step-test* consiste em aplicar, no mesmo espécime, níveis crescentes de carga (*steps*) por um número fixo de ciclos (exemplo 10.000 ciclos) até a ocorrência de falha do material. O ensaio inicia-se com a aplicação de uma carga inferior a resistência máxima do material, por um número fixo de ciclos. Se o espécime sobreviver, aumenta-se a carga em um incremento fixo e o espécime é ciclado novamente. Esse procedimento é repetido até a fratura da amostra, momento em que se registra a carga para fratura e o número de ciclos (COLLINS, 1993). Os dados são então analisados por meio de análise de sobrevivência de Kaplan Meier (COLLINS, 1993; NICOLAISEN et al., 2014; MAGNE et al., 2015) ou estatística de Weibull (COLLINS, 1993; ANAMI et al., 2016). O método de aplicação de carga em *steps* crescentes também tem sido utilizado para obtenção de informações sobre a confiabilidade de um tratamento restaurador em ensaios com menor tempo de duração (BASSO et al., 2016; SHEMBISH et al., 2016). Nesses estudos, três perfis de tensão (suave, moderado e agressivo) são geralmente empregados para distribuir as falhas entre os diferentes *steps* de carga, permitindo uma melhor predição clínica (BONFANTE; COELHO, 2016).

No cenário atual, diversos estudos têm utilizado os métodos *staircase* e *step-test* para investigar o comportamento à fadiga das cerâmicas, com variações no tempo de vida (número máximo de ciclos) e na frequência de aplicação de carga. A descrição de alguns desses estudos pode ser encontrada nas Tabelas 1 e 2.

Tabela 1 – Descrição de estudos que avaliaram o comportamento à fadiga dos materiais cerâmicos pelo método *staircase*.

(continua)

<table>
<tr><th>Autores (ano)</th><th>Objetivo</th><th>Frequência</th><th>Número de ciclos</th><th>Desfecho (resistência à fadiga; carga para falha em fadiga) - Média (desvio-padrão)</th></tr>
<tr><td>Polli et al. (2016)</td><td>Avaliar o efeito do desgaste (com e sem refrigeração) na resistência à fadiga de barras de Y-TZP (ensaio de flexão quatro pontos).</td><td>10 Hz</td><td>500.000</td><td>Y-TZP controle (apenas sinterizada): 448 (56) MPa
Y-TZP desgaste sem refrigeração: 520 (34) MPa
Y-TZP desgaste com refrigeração: 510 (27) MPa</td></tr>
<tr><td>Amaral et al. (2016)</td><td>Avaliar o efeito do jateamento e da aplicação de glaze na resistência à fadiga de discos de Y-TZP (ensaio de flexão biaxial).</td><td>0,5 Hz</td><td>100
1.000
10.000
100.000</td><td><table>
<tr><th></th><th>100 ciclos</th><th>1.000 ciclos</th><th>10.000 ciclos</th><th>100.000 ciclos</th></tr>
<tr><td>Y-TZP (controle)</td><td>817 (59) MPa</td><td>784 (52) MPa</td><td>769 (79) MPa</td><td>777 (26) MPa</td></tr>
<tr><td>Y-TZP jateada</td><td>1147(19) MPa</td><td>1096(39) MPa</td><td>1072(27) MPa</td><td>1023(122) MPa</td></tr>
<tr><td>Y-TZP glaze</td><td>921 (47) MPa</td><td>835 (29) MPa</td><td>826 (64) MPa</td><td>799 (14) MPa</td></tr>
</table></td></tr>
<tr><td>Dibner e Kelly (2016)</td><td>Avaliar o efeito de diferentes espessuras de cerâmica de infraestrutura (dissilicato de lítio) e de cobertura (cerâmica vítrea de nano-fluorapatita) na carga para fadiga de discos cimentados em bases de resina epóxica.</td><td>20 Hz</td><td>500.000</td><td>Apenas dissilicato (1,5 mm): 501 (70) N
Dissilicato (1 mm) e cobertura (0,5 mm): 538 (42) N
Dissilicato (0,75 mm) e cobertura (0,75 mm): 600 (133) N
Dissilicato (0,5 mm) e cobertura (1 mm): 611 (130) N</td></tr>
<tr><td>Pereira et al. (2016)</td><td>Avaliar o efeito do desgaste com brocas diamantadas na resistência à fadiga de discos Y-TZP (ensaio de flexão biaxial).</td><td>6 Hz</td><td>20.000</td><td>Y-TZP (Lava Frame) apenas sinterizada: 577 (57) MPa
Y-TZP (Lava Frame) desgastada: 706 (62) MPa
Y-TZP (Zirlux FC) apenas sinterizada: 542 (25) MPa
Y-TZP (Zirlux FC) desgastada: 652 (83) MPa</td></tr>
<tr><td>May et al. (2015)</td><td>Avaliar o efeito da espessura oclusal de cimento resinoso na carga para a fadiga de coroas feldspáticas confeccionadas por CAD/CAM.</td><td>20 Hz</td><td>500.000</td><td>Coroa feldspática, 50 µm de cimento: 246 (23) N
Coroa feldspática, 500 µm de cimento: 159 (23) N</td></tr>
</table>

(conclusão)

Autores (ano)	Objetivo	Frequência	Número de ciclos	Desfecho (resistência à fadiga; carga para falha em fadiga) - Média (desvio-padrão)
Belli et al. (2014)	Comparar a resistência à fadiga entre diferentes materiais cerâmicos e resinas compostas (ensaio de flexão quatro pontos).	0,5 Hz	20.000	Y-TZP: 440 (51) MPa Dissilicato de lítio para CAD/CAM: 121 (15) MPa Feldspática para CAD/CAM: 38 (2) MPa Resina composta CAD/CAM: 64 (6) MPa

Tabela 2 – Descrição de estudos que avaliaram o comportamento à fadiga dos materiais cerâmicos pelo método *step-test*.

Autores (ano)	Objetivo	Frequência	Protocolo de *steps*	Desfecho (resistência à fadiga; carga para falha em fadiga; número de ciclos) Média (desvio-padrão)
Anami et al. (2016)	Investigar o efeito de tratamentos de superfície na carga para fratura em fadiga de coroas Y-TZP recobertas por cerâmica feldspática cimentadas a resina epóxica.	1,4 Hz	200 N – 5.000 ciclos 800 N – 10.000 ciclos 1000 N – 10.000 ciclos 1200 N – 10.000 ciclos 1400 N – 10.000 ciclos Máximo: 45.000 ciclos	Y-TZP sem tratamento: 1093 (237) N Y-TZP jateamento com 125 µm Al_2O_3: 973 (198) N Y-TZP jateamento com 30 µm SiO_3: 920 (147) N Y-TZP glaze: 867 (180) N
Magne et al. (2015)	Avaliar a carga para fadiga de coroas ultrafinas (0,7 mm de espessura no sulco central) confeccionadas por usinagem CAD/CAM e cimentadas adesivamente a dentes molares.	10 Hz	200 N – 5.000 ciclos 400 N – 30.000 ciclos 600 N – 30.000 ciclos 800 N – 30.000 ciclos 1000 N – 30.000 ciclos 1200 N – 30.000 ciclos 1400 N – 30.000 ciclos Máximo: 185.000 ciclos	Dissilicato de lítio: 1123 (130) N Feldspática: 987 (200) N Resina composta: 1014 (146) N
Nicolaisen et al. (2014)	Comparar a resistência à fadiga de coroas metalocerâmicas e coroas de Y-TZP recobertas com cerâmica vítrea.	12 Hz	400 N – 600.000 ciclos 600 N – 200.000 ciclos 800 N – 200.000 ciclos 1000 N – 200.000 ciclos Máximo: 1.200.000 ciclos	Coroa metalocerâmica: 772.000 (133.000) ciclos Coroa totalmente cerâmica: 785.000 (202.000) ciclos

2.6.1.1 Variáveis envolvidas em ensaios de fadiga cíclica

A condução de ensaios de fadiga cíclica envolve a determinação prévia de algumas variáveis, como o número máximo de ciclos e a frequência de aplicação de carga (número de ciclos realizados por segundo).

Até o momento, não há um consenso sobre o número de ciclos empregado no ensaio de fadiga e o tempo em serviço correspondente. Segundo Wiskott, Nicholls e Belser (1995), para apresentar relevância clínica, os testes de fadiga deveriam ser realizados com, no mínimo, 10^6 ciclos, valor equivalente a um ano em boca. Para chegar a esse número, os autores assumiram 3 períodos de 15 minutos de mastigação por dia, a uma taxa de 60 ciclos por minuto (1 Hz). Assim, em um dia, ter-se-iam 2700 ciclos, resultando em aproximadamente 10^6 ciclos por ano. Entretanto, salienta-se que esses achados são baseados em uma simplificação matemática, não sendo suportados por evidência clínica.

Muitos testes de resistência à fadiga de materiais cerâmicos têm sido conduzidos com frequências entre 1 Hz e 2 Hz, tendo-se em vista que essa é uma aproximação da frequência do ciclo mastigatório (PO et al., 2011). Entretanto, a utilização de frequências baixas pode tornar a coleta dos dados muito lenta. Conduzindo-se o teste de fadiga a uma frequência de 2 Hz e utilizando-se 10^6 ciclos, seriam necessários, para um único corpo de prova, aproximadamente 6 dias para se concluir a ciclagem mecânica. Caso uma frequência superior, como 20 Hz, fosse utilizada, esse tempo se reduziria para 14 horas, o que otimizaria a coleta dos dados.

Estudos têm sido realizados para se verificar o efeito do aumento da frequência na resistência e nos parâmetros de fadiga de diferentes materiais.

Rosentritt et al. (2006) avaliaram a carga para fratura de próteses parciais fixas de três elementos confeccionadas de dissilicato de lítio após envelhecimento com ciclagem mecânica realizada com frequência de 1,6 Hz e 3 Hz, empregando-se 1.200.000 ciclos e 50 N de carga. Não houve diferença significativa na carga para fratura após ciclagem com 1,6 Hz (1300 N ± 557 N) e 3 Hz (1350 N ± 487 N).

Kelly et al. (2010) investigaram o efeito da frequência (2, 10 e 20 Hz) na carga para fratura em fadiga de discos de cerâmica aluminizada infiltrada por vidro cimentados com fosfato de zinco a bases de um material compósito análogo a dentina, utilizando o método da escada. As cargas de fratura para as frequências de 2 Hz, 10 Hz e 20 Hz foram 569,64 N, 603,13 N e 602,5 N, respectivamente. Esses resultados apontam para um pequeno aumento na carga para fratura em fadiga quando utilizada frequência de 20 Hz, em comparação aos resultados obtidos com frequência de 2 Hz.

Joshi et al. (2014) avaliaram o efeito da frequência no tempo para falha em uma cerâmica vítrea de fluorapatita. O ensaio foi realizado em configuração de flexão uniaxial quatro pontos, na presença de água deionizada a 37ºC e carregamento sinusoidal com aplicação de tensão máxima entre 45 MPa e 60 MPa. As frequências investigadas foram 2 Hz e 10 Hz. O tempo de vida característico para os corpos de prova ensaiados a 2 Hz foi de 10.800 segundos (IC 95%: 2.800 - 40.300 segundos), enquanto para o grupo de 10 Hz foi de 11.600 segundos (IC 95%: 1.800 - 87.900 segundos). Desse modo, a aceleração do ensaio, por meio do emprego de maior frequência, não resultou em economia de tempo.

Fukushima (2015) não encontrou diferenças significativas no tempo de vida característico obtido para a uma cerâmica Y-TZP quando da realização do ensaio de fadiga cíclica com 2 Hz (967.602 segundos; IC 95%: 19.105 - 49.000.000 segundos) e 10 Hz (4.068 segundos; IC 95%: 290 - 56.980 segundos). O parâmetro de crescimento subcrítico de trinca (n) foi de 48 para 2 Hz e 40 para 10 Hz.

O efeito da frequência de aplicação de carga na resistência à fadiga pode ser influenciado pelo ambiente de realização do ensaio. Segundo Souza (1982), para materiais metálicos, os valores de resistência à fadiga obtidos em ensaios realizados em ambientes corrosivos, ao contrário dos ensaios ao ar, são dependentes da velocidade do ensaio, sendo que quanto mais alta for essa velocidade, menor será a redução do limite de fadiga devido a corrosão.

3 ARTIGO 1 – *IMPACT OF THE CAD/CAM MACHINING ON THE FATIGUE BEHAVIOR OF GLASS AND POLYCRYSTALLINE CERAMICS*

Este artigo será submetido ao periódico *Dental Materials*, *Elsevier*, ISSN: 0109-5641, Fator de impacto = 3.931; Qualis A1. As normas para publicação estão descritas no Anexo A.

Impact of the CAD/CAM machining on the fatigue behavior of glass and polycrystalline ceramics

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ABSTRACT

Objective: to assess the effect of CAD/CAM machining on the biaxial flexural fatigue strength and on the surface roughness of different ceramics by comparing machined vs. machined and polished specimens.

Methods: disc shaped specimens of Y-TZP, leucite- and lithium disilicate-based glass ceramics were prepared by CAD/CAM machining, and assigned into two groups: machining (M); and machining followed by polishing (MP). The surface roughness was measured and the biaxial flexural fatigue strength was evaluated by the step-test method (n=20). The initial load and the load increment for each ceramic material were based on a monotonic test (n=5). A maximum of 10,000 cycles was applied in each load step, at 1.4 Hz. Weibull probability statistics was used for the analysis of the biaxial flexural fatigue strength, and Mann-Whitney test (α=5%) to compare roughness between M and MP condition.

Results: M resulted in lower values of characteristic flexural fatigue strength (FFS) than MP. The greatest reduction in the FFS from MP to M was observed in Y-TZP (40%; M=536.48 MPa; MP=894.50 MPa), followed by lithium disilicate (33%; M=187.71 MPa; MP=278.93 MPa) and leucite glass ceramic (29%; M=72.61 MPa; MP=102.55 MPa). Significantly higher values of roughness (Ra) were observed for M than for MP (leucite: M=1.59 µm and MP=0.08 µm; lithium disilicate: M=1.84 µm and MP=0.13 µm; Y-TZP: M=1.79 µm and MP=0.18 µm).

Significance: machining affected negatively the biaxial flexural fatigue strength of CAD/CAM ceramics. The results suggest that machining of partially sintered ceramics may be as deleterious as machining of fully sintered ceramics.

KEYWORDS: Computer-Aided Design; Computer-Aided Machining; Fatigue; Biaxial Flexural Strength; Roughness; Zirconia; Leucite Glass Ceramic; Lithium Disilicate.

HIGHLIGHTS

1) The effect of machining on flexural fatigue strength of CAD/CAM ceramics was evaluated.
2) Machining reduces the fatigue strength of CAD/CAM ceramics.
3) Y-TZP showed the greatest reduction in the fatigue strength due machining.
4) Soft machining may be as deleterious as hard machining.
5) Higher values of roughness may be expected after machining.

1 Introduction

The improvements on the mechanical properties of dental ceramic systems and the implementation of CAD/CAM (Computer-Aided Design; Computer-Aided Machining) technology in restorative dentistry have been contributing to widely increase the use of all-ceramic restorations as an alternative to satisfy the high esthetic demand of the patients [1].

A great variety of ceramic materials are available for CAD/CAM, which differ in terms of microstructure, mechanical behavior and also regarding machining mode [2,3]. Feldspar-, leucite- and lithium disilicate-based ceramics are commonly available in a full sintered stage for hard machining [4]. These materials contain a high volume of glassy phase and, consequently, lower fracture toughness than polycrystalline ceramics [5,6]. In lithium disilicate glass ceramics, the presence of lithium disilicate crystals promote crack deflection, improving its fracture strength [7]. Therefore, this material is indicated for the manufacture of veneers, inlays, onlays, anterior and posterior crowns, such as leucite glass ceramics [8], but also for implant superstructures of single-tooth restorations [9].

Yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP) is commonly milled in a partially sintered stage, in a process called soft machining, and after that, it is subjected to high temperature for full sintering [4]. Y-TZP ceramics exhibit a transformation toughening mechanism that acts to resist crack propagation. It involves the transformation of metastable tetragonal crystallites to the monoclinic phase at the crack tip, which, accompanied by a volumetric expansion, induces compressive stresses, hindering further crack propagation [10]. It may be used as infrastructure for fixed partial dentures (FDPs) covered by glass-ceramic [3] or as monolithic full-contour restorations [11,12], besides being indicated for implant abutments [3].

Despite of promising clinical success rates, framework fracture of all-ceramic restorations is still a technical problem [13–15], which may demand the replacement of the restoration with additional costs to the patient and to the dentist. In a recent systematic review, with the inclusion of 67 clinical studies, 5-year framework fracture rates of up to 18.4%, 5.5% and 1.7% were reported for single crowns made of feldspathic ceramic, reinforced glass ceramic and densely sintered zirconia, respectively [14]. When multiple-unit fixed dental prostheses were considered, these rates increased up to 15.3% for reinforced glass ceramic and up to 3.2% for densely sintered zirconia [15].

Due to the brittle nature of ceramics, their fracture strength is strongly influenced by the presence of defects, which can be considered especially critical when located at zones of tensile stress concentration [16,17]. The fractography analysis of clinically failed all-ceramic restorations [16,18,19] and the finite element analysis [20,21] showed that the cementation surface of all-ceramic crowns concentrates tensile stress and that defects on this surface may be the origin of fracture in failed restorations [16,18,19]. Therefore, procedures that affect the intaglio surface of all-ceramic restorations must be investigated regarding their impact on the ceramic strength.

In spite of reducing processing defects, once the CAD/CAM blocks are produced in a standard process, the machining induces a complex network of events in the ceramic, resulting in radial and lateral cracks, chipping, subsurface damage, residual stresses and even plastic deformation [22–24]. Hard machining reduced in around 27% the biaxial flexural strength of a leucite glass ceramic, inducing damage on the ceramic surface [25]. Soft machining resulted in surface damage and significantly reduced the strength of zirconia, which may result in unexpected failures at stresses much lower than the ideal strength of the material [26]. Kelly et al. (1991) [27] reported that the defects introduced by the CEREC® CAD/CAM system seemed to be the origin of the failure in ceramics subjected to a uniaxial bending test.

Besides, it is important to consider that in the oral environment all-ceramic restorations are subjected to many challenges, such as cyclic loads, humidity, pH and temperature variations. Consequently, they trend to fail due fatigue [28]. Ceramics are susceptible to a slow and stable crack growth (SCG) when subjected to stresses below the critical value, especially in the presence of water. This phenomenon can eventually lead to strength degradation over time, decreasing the lifetime of dental prostheses and seems to be most related to the ceramic microstructure [29].

Therefore, considering the increasing use of CAD/CAM technology in restorative dentistry, in which machining may introduce new features to the cementation surface of ceramic restorations, and the susceptibility of ceramic materials to the fatigue process, this study aimed to assess the effect of CAD/CAM machining in the biaxial flexural fatigue strength and in the surface roughness of a leucite-based glass ceramic, a lithium disilicate-based glass ceramic and a yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP). The experimental hypothesis tested was that, for all ceramics investigated, machining would result in lower values of biaxial flexural fatigue strength and in higher values of roughness when compared to polishing after machining.

2 Materials and methods

Table 1 describes the materials used in this study.

2.1 Machining of ceramic discs by CAD/CAM

Disc-shaped ceramic specimens were prepared by machining in a CEREC inLab MC XL milling unit (Sirona Dental Systems Gmbh, Germany), according to the methodology described by Fraga et al. (2015) [25] (Fig. 1), using three different ceramic materials: leucite-based glass ceramic (IPS Empress CAD, C14, Ivoclar Vivadent AG, Liechtenstein), milled in a fully-sintered stage; lithium disilicate-based glass ceramic (IPS e.max CAD, LT, C14, Ivoclar Vivadent AG, Liechtenstein), milled in a pre-crystallized stage ("blue stage"); and Y-TZP (Vita In-Ceram YZ, Vita Zahnfabrik, Germany), milled in a partially-sintered stage.

Two pairs of diamond burs, each one containing one cylindrical (Cylinder Pointed Bur 12S and Cylinder Pointed Bur 20, Sirona Dental Systems Gmbh, Germany) and one stepped pattern bur (Step Bur 12S and Step Bur 20, Sirona Dental Systems Gmbh, Germany) were used in the machining process of each type of ceramic material (Table 1). Fifty discs were obtained from lithium disilicate and from Y-TZP blocks, and the set of burs was changed after the machining of 25th disc. Forty discs were obtained from leucite-based glass ceramic material, and the set of burs was changed after the machining of the 20th disc. The machining order and the pair of bur used were recorded for each individual disc and the effect of these variables on the surface roughness and on the flexural fatigue strength was evaluated afterwards.

Each block of leucite (12.0 mm X 14.0 mm X 18.0 mm) and lithium disilicate (12.4 mm X 14.5 mm X 18.0 mm) resulted in one disc, while each block of Y-TZP (15.5 mm X 19 mm X 39 mm) resulted in two discs. Immediately after machining, leucite and lithium disilicate discs had about 13.5 mm diameter and 1.4 mm thickness, while no-sintering Y-TZP discs had about 18 mm diameter and 1.8 mm thickness. The Y-TZP discs were bigger than the others because the software used in the machining process (CEREC inLab 3D, version 4.0, Sirona Dental Systems Gmbh, Germany) automatically compensated the sintering shrinkage expected for zirconia materials (approximately 20-25%).

2.2 Experimental groups

After machining, the discs of each ceramic material were randomly assigned into two groups, using the software Random Allocator (www.random.org): 1) machining (M), and 2) machining followed by polishing (MP). Therefore, the present study consisted in six experimental groups, assigned according to the material (leucite glass ceramic, lithium disilicate and Y-TZP) and the treatment performed on the bottom surface of the discs (machining - M, and machining followed by polishing - MP).

In the MP groups, polishing was performed manually under water cooling, on the bottom surface of the discs, using 400, 600 and 1200 grit silicon carbide paper for leucite and lithium disilicate and 1200 grit silicon carbide paper for Y-TZP. It was removed a thickness of 80 µm of the machined surface, in order to eliminate the irregularities introduced by machining [24,25]. A micrometer (210 MAP, Starrett, USA) was used to control the depth of removal.

The final thickness of all the specimens was adjusted on the upper surface of the discs, using 240, 400, 600 and 1200 grit silicon carbide paper for leucite and lithium disilicate glass ceramics, and 1200 grit silicon carbide paper for Y-TZP.

After these procedures, lithium disilicate specimens were submitted to heat treatment in a Vita Vacumat 6000 MP furnace (Vita Zahnfabrik, Germany) to promote full crystallization and Y-TZP specimens were densely sintered in Vita Zyrcomat furnace, following the protocols indicated by the manufacturers. The final diameter of the discs was 13.5 mm, and the final thickness was 1.31 ± 0.01 mm for leucite glass ceramic, 1.30 ± 0.01 mm for lithium disilicate, and 1.29 ± 0.01 for Y-TZP discs.

2.3 Measurement of the surface roughness

The roughness of the bottom surface of each disc was measured prior the mechanical tests, using a contact stylus profilometer (SJ-410, Mitutoyo, Japan).

The Ra (average surface roughness; µm) and Rz (the arithmetic mean peak-to-valley height; µm) values were determined using the average of three measurements, transversal to the machining path. The sampling length was equivalent to five times the cut-off value (λc), as defined according to ISO 4287:1997 [30], using the Ra values recorded at a first reading as a reference. Thereafter, the roughness of the MP leucite glass ceramic specimens was evaluated using λc=0.25 mm (tabulated value for 0.02<Ra≤0.1 mm), resulting in a sampling length of 1.25 mm. The roughness of the other groups was measured using λc=0.8 mm (tabulated value for 0.1<Ra≤2.0 mm), resulting in a sampling length of 4 mm. In addition to the use of the correct cut-off value, a Gaussian filter was employed to differentiate between shape defects and roughness profile.

2.4 Monotonic biaxial flexural strength

Prior to fatigue test, the monotonic biaxial flexural strength was determined for lithium disilicate and Y-TZP specimens, in both conditions: machining (M) and machining followed by polishing (MP) (n=5). For the leucite groups (M and MP), the data regarding the monotonic biaxial flexural strength were taken from Fraga et al. (2015) [25].

The test was performed using a piston-on-three ball assembly, according to the ISO 6872:2008 [31], in a universal testing machine (DL-1000 Emic, Brazil). The bottom surface of the disc – refers to the M or MP surface – was positioned on the top of three steel spheres (2.5 mm in diameter, 120º apart, and forming a circle of 10 mm diameter), with a load applied at a rate of 1 mm/min, perpendicular to the center of the top surface of the disc, by a circular cylinder steel piston with a 1.4 mm diameter flat tip. The fracture strength, in MPa, was calculated using Eqs. (1)–(3) [31].

$$\sigma_m = -0.2387P(X - Y)/b^2 \quad \text{Eq. (1)}$$

$$X = (1 + \nu)\ln(B/C)^2 + [(1 - \nu)/2](B/C)^2 \quad \text{Eq. (2)}$$

$$Y = (1 + \nu)\left[1 + \ln A/C^2\right] + (1 - \nu)(A/C)^2 \quad \text{Eq. (3)}$$

where P is the load at fracture (N), b is the disc thickness (mm), A is the support ball radius (5 mm), B is the radius of the tip of the piston (0.7 mm), and C is the specimen radius (6.75 mm). A specific Poisson ratio (ν) was used for each ceramic material (0.20 for the leucite glass-ceramic; 0.21 for the lithium disilicate glass-ceramic; 0.26 for the Y-TZP), based on the mean values obtained in the Resonant Ultrasound Spectroscopy (RUS) methodology described by Belli et al. (2016) [32].

2.5 Flexural fatigue test

The biaxial flexural fatigue strength of the groups (n=20) was evaluated by means of the step-test method in an adapted fatigue tester (Fatigue Tester, ACTA, The Netherlands) using the same piston-on-three ball configuration [31] in water.

The step-test consists in to subject the specimen to a prescribed number of cycles at each of a series of increasing stress levels, until the failure of the specimen. To start a step-test, a stress level below to the expected material fatigue strength is selected. The specimen is then tested at that stress level until either failure occurs or the run-out at a previously set number of cycles is achieved. If failure occurs, the stress level and the number of cycles are recorded. If run-out occurs, the stress level is increased by a preselected stress increment and the same specimen is run again at the new stress level. Again, if the specimen fails, the data are recorded; and if run-out occurs, the stress level is again increased for a new run using the same specimen. This procedure is continued until the specimen does fail [33].

Cyclic load was applied at a frequency of 1.4 Hz. The maximum number of cycles at each load step was set in 10,000 cycles. If the specimen survived the 10,000 cycles, the stress level was increased by a fixed load increment in the same specimen. An individual loading protocol was established for each type of ceramic based on the results of the monotonic test. For leucite glass ceramic groups, the cycling started with 30 N. If the specimen survived the 10,000 cycles, the load was increased a step of 10 N and, then, successively until the failure of the specimen. In lithium disilicate groups, it was applied an initial load of 60 N and a step of 20 N, while for Y-TZP specimens it was used an initial load of 300 N and a step of 50 N. The cycling was continued until the failure of the specimen.

The software was adjusted to register the load and the number of cycles corresponded to the fracture of the specimen.

The maximum biaxial flexural fatigue load (L_E) supported by each specimen was calculated according to the equation described by Nicholas (2006) [34] (Eq. 4):

$$L_E = L_0 + \Delta L \left(\frac{N_{fail}}{N_{life}} \right) \quad \text{Eq. (4)}$$

where L_0 is the previous maximum biaxial flexural fatigue load that did not result in failure, ΔL is the load step increase, N_{fail} is the number of cycles to failure at the failure load step ($L_0 + \Delta L$), and N_{life} is the defined cyclic fatigue life (10,000 cycles). The linear interpolation concept embodied in Eq. (4) considers that damage accumulation might be a linear function of cycles [34]. Equations (1) – (3) were used to calculate the biaxial flexural fatigue strength value, in MPa, correspondent to L_E for each specimen.

2.6 Field emission scanning electron microscopy analysis (FESEM)

To evaluate the effectiveness of the polishing protocols in removing the machining damage, one extra disc of each group was analyzed in a field emission scanning electron microscope (FESEM) (Inspect F50, FEI, USA). To assess the cross-sectional surface of the machined discs (M groups) and of the machined and polished discs (MP groups), the extra samples were included in acrylic resin and cut, perpendicularly to the M and MP surfaces, using a diamond saw at low speed (Isomet 1000, Buehler, Lake Buff, USA). After cutting, the samples were detached from the acrylic resin and had their cross-sectional surfaces polished (400, 600, 800, and 1200 grit silicon carbide paper) and sputter-coated with gold-palladium for FESEM.

2.7 Statistical analysis

Weibull probability statistics [35,36] was used for the analysis of the biaxial flexural fatigue strength, as indicated by the characteristic flexural fatigue strength (σ_0), which is the flexural fatigue strength occurring at a probability of failure of 63.2% for a particular test specimen, and Weibull modulus (m), which represents the reliability of the fracture strength. The basic form of the Weibull distribution is shown in Eq. (5) [37]:

$$P_f = 1 - \exp[-(\sigma/\sigma_0)^m] \qquad \text{Eq. (5)}$$

where P_f is the probability of failure and σ is the failure stress. The biaxial flexural fatigue strength data were ranked in ascending order and the probability of failure for each failure stress ($P_f(\sigma_i)$) was calculated by Eq. (6), where i is the rank order (1, 2, 3…N) and N is the number of specimens per group (n = 20) [37].

$$P_{f\sigma i} = (i - 0.5)/N \qquad \text{Eq. (6)}$$

A rank regression was performed on the biaxial flexural fatigue strength data to estimate the Weibull parameters (σ_0 and m) [31]. The upper and lower limits of the 95% confidence intervals for σ_0 and m were calculated according to ENV 843-5:1996 [38]. Differences were considered significant when confidence intervals failed to overlap.

The non-parametric Mann-Whitney test ($\alpha = 0.05$) was used to compare the roughness values (Ra and Rz) between M and MP groups for each ceramic material, since data presented heterogeneity of variances ($p<0.05$ on the Levene test).

Using the data of the M groups (n = 20), the roughness and the biaxial flexural fatigue strength obtained for each set of burs were compared by means of the Student's t-tests ($\alpha = 0.05$). Spearman correlation coefficients (r_s) ($\alpha = 0.05$) were calculated to investigate the influence of the machining order in the roughness surface and in the biaxial flexural fatigue strength for each type of ceramic material, considering the set of burs separately.

3 Results

The step-load profiles of all ceramic materials showed MP groups failing under higher load ranges than M groups, as indicated in Fig. 2. The machining (M) resulted in significantly higher values of roughness and lower values of characteristic flexural fatigue strength when compared to the machining followed by polishing groups (MP) (Table 2). The greatest reduction in the characteristic flexural fatigue strength from MP to M was observed in Y-TZP (40%), followed by lithium disilicate (33%) and leucite glass ceramic (29%). Polishing reduced the roughness in approximately 94%, 92% and 85% for leucite, lithium disilicate and Y-TZP, respectively. The Weibull modulus was similar for the conditions M and MP in all ceramics investigated.

The FESEM images indicate that machining induced defects in all ceramics investigated and that polishing was effective in removing these defects (Fig. 3).

Regarding the pair of burs used in the machining, Student's t-test showed differences in the roughness values between the two set of diamond burs used in the machining of leucite glass-ceramic and Y-TZP discs. The fatigue strength of Y-TZP samples was also significantly influenced by the pair of burs (Table 3).

The correlation between machining order and roughness/flexural fatigue strength depending on the ceramic material and on the set of burs (Table 3). For leucite and lithium disilicate specimens, Spearman coefficient indicated a significant and high [39] correlation between machining order and Ra just for one of the two set of burs used in the machining. Regarding machining order and flexural fatigue strength, a significant and high [39] correlation was found in leucite and Y-TZP specimens for just one of the set of burs.

4 Discussion

In the present study, CAD/CAM machining (M groups) introduced defects in the ceramic surface (Fig. 3), increasing surface roughness and significantly reducing the characteristic flexural fatigue strength of all ceramics investigated, when compared to the groups where surface defects were removed by polishing (MP groups). Therefore, the experimental hypothesis was accepted.

The knowledge about how much CAD/CAM machining affects the mechanical behavior of ceramics with different microstructures is very important since the fracture strength of these materials is sensitive to the presence of defects [17]. These defects can be considered especially critical when located at the cementation surface of a ceramic crown due to the concentration of tensile stress in this area, which may contribute to initiate the clinical fracture of an all-ceramic restoration [16]. The cervical margin of a crown may also be considered a critical zone. As it usually presents a thinner structure, difficult for bur access, defects may be introduced by machining contributing to initiate the failure [40]. Concerning authors knowledge, despite CAD/CAM technology has been widely used in restorative dentistry, this is the first study that assessed the effects of CAD/CAM machining on the fatigue behavior of ceramics with different microstructures.

Leucite- and lithium disilicate-based glass ceramics are available for hard machining process, which implies that the restoration is milled from an industrial densely sintered block [1], and may require higher removal forces in machining [41]. Lithium disilicate ceramics are machined in the “blue stage”, prior to full crystallization, which contains lithium metasilicate (Li_2SiO_3) crystals. In this stage, the ceramic is easy to machine [1] and exhibits moderate biaxial strength (130 MPa) [42]. After machining, the restorations are submitted to a heat treatment to ensure final crystallization into lithium disilicate, and to have its flexural strength improved (360 MPa) [42]. This heat treatment is likely to reduce the extend of the residual stresses from machining and ensures the development of a microstructure composed of interlocked lithium disilicate crystals [4]. However, the damage introduced by machining was still present after the heat treatment, as seen in FESEM images (Fig.3), negatively affecting the characteristic fatigue strength of the material.

Y-TZP restorations are commonly milled from partially sintered blocks, in a process called soft machining, and then sintered at high temperature. The restoration is milled in a bigger size to compensate the volume shrinkage that will occur during sintering (about 25%) [43]. In the present study, the greatest difference between MP and M groups was seen in Y-

TZP ceramic, milled in a partially sintered stage, where machining reduced in 40% the material characteristic flexural fatigue strength. The reduction in the characteristic fatigue strength was around 33% for lithium disilicate and 29% for leucite glass ceramic. Therefore, an important finding of this study was that, in spite of partially sintered blocks been softer and easier to mill, soft machining may also introduce damage in the ceramic surface, being as deleterious for the material's flexural fatigue strength as hard machining. Besides of this, the heat treatment performed in Y-TZP to complete sintering seemed not to be able to eliminate all the machining defects, which agrees with Kelly and Rungruanganunt (2016) [44], which found severe machining damage in zirconia implant abutments.

To assess the effect of the machining in the fatigue behavior of ceramics with different microstructures, one group of each material (MP group) was submitted to a polishing procedure after machining, which removed 80 µm from the machined surface, ensuring that defects from machining were removed. Few studies have investigated the deep of the damage introduced by machining in the ceramic surface, and the reported values ranged from 9 µm [27] to 60 µm [24] in glass ceramics. Therefore, we believe that the polishing protocol adopted in the present study, with a reduction of 80 µm of the machined surface, was effective in removing the defects introduced by machining, as it can be seen in the FESEM and in the roughness analysis (Fig.3, Table 2).

The literature reports Ra values after machining in a CEREC inLab unit of 1.37 µm [25] and 1.47 µm [45] for a leucite-based glass ceramic, and of 1.53 µm and 1.20 µm for Y-TZP samples [46]. Roughness after machining may be influenced by the material hardness and by the grit size of the tool [47]. The pattern of peaks and valleys introduced by machining with diamond burs was reported to be deeper for a soft material (a resin nanoceramic composite) than for hard materials (fine particle feldspar ceramic; resin-infiltrated ceramic)

[47]. Special care should be given to selecting tool grit size when soft materials are milled to optimize the peaks and valleys of the milled surface.

Weibull modulus, a shape parameter that describes the relative spread of strength values in the asymmetrical distribution [37], did not differ significantly between M and MP conditions for each ceramic (Table 2). However, in the step-load profile graphs (Fig.2) is possible to notice that polishing resulted in a greater scatter of the data. A possible explanation for this finding is that polishing was performed manually, without load and speed control, which may contribute increase the variability in the flexural fatigue values of the MP groups.

The step-test method can be considered a versatile fatigue test. It consists in a time-varying load test, in which the specimens are subjected to successively higher stress levels in a predetermined number of cycles [48]. This methodology has been employed to investigate the fatigue resistance of restorative treatments, and the data are submitted to survival analysis, such as Kaplan Meier [49–51]. According to Collins (1993) [33], when the step-test data have been acquired, they may be plotted on a probability plot, from which the mean fatigue strength and the standard deviation may be determined for the defined cyclic fatigue life (number of cycles in an individual load step). Considering that strength data of brittle materials typically fit in the Weibull distribution [37], in the present study, the step-test data where plotted on a Weibull probability paper, and the values of characteristic fatigue strength and Weibull modulus calculated for a life time of 10,000 cycles. The disadvantage of this method lies on the unknown effects of latent damage that may be incurred while stepping up through the series of stress levels to reach the failure stress levels [33]. More recently, step-test has also been used to obtain reliability information (e.g., mean life, probability of failure at a specific time) about a restorative treatment in a shorter time [52,53]. In these studies, three stress profiles (mild, moderate, and aggressive) are usually employed to distribute failure across different step loads and to allow better statistic predictions [54].

The comparison of the mean strength values obtained in the monotonic and in the fatigue test (Table 2) showed that fatigue test resulted in lower values of flexural strength. The reduction on the material strength trended to be higher for glass-ceramic groups (around 33% to 44%) than for Y-TZP groups (around 15%). Despite of showing the lowest strength degradation when compared to monotonic results, Y-TZP ceramic was the most affected by machining. A previous study [55] indicated a strength degradation (difference between monotonic and fatigue strength) of about 42.7% for Y-TZP, 53.4% for a CAD/CAM lithium disilicate, and 45.5% for a feldspathic ceramic. The microstructure significantly influenced the slow crack growth behavior of the dental ceramics [6,56]. Polycrystalline ceramics presented low susceptibility to slow crack growth and low degradation over time, while porcelains showed the lowest values of resistance to crack propagation, resulting in high strength degradation over time [6]. These findings support the trend evidenced in the present study.

Regarding the burs used for machining, in the present study significant differences were found in terms of roughness and fatigue strength between the two set of burs used in the machining of Y-TZP specimens (Table 3). Differences in the Ra values and in the biaxial flexural strength were reported in a study that employed six pair of burs to machining feldspathic ceramic discs in a CEREC inLab MC XL® unit [57]. Different set of burs also influenced que roughness of Y-TZP specimens, but did not affect the reliability of the Y-TZP restorations [46]. The correlation between machining order and roughness/flexural fatigue strength was also dependent on the set of burs used in the machining. Therefore, considering that the set of burs may influence the roughness and the strength of the ceramic, special care should be taken in future studies involving CAD/CAM machining to randomly distribute among the experimental groups specimens milled by different set of burs, avoiding any possible interference related to the diamond milling tools on the results.

For leucite and Y-TZP materials, Spearman correlation coefficients indicated that, for a maximum of 20 machined specimens, a possible deterioration of the machining burs may have no deleterious effect on the ceramic fatigue strength, since a positive correlation between machining order vs. fatigue strength means that increasing machining order may increase the flexural fatigue strength. The correlation between machining order vs. roughness was significant and negative for leucite, which means a trend for decreasing roughness with increasing machining order. However, for lithium disilicate a positive correlation was found, which means that increasing machining order may increase the roughness surface. Nevertheless, the increased roughness did not impact in the fatigue strength, since no significant correlation was found between machining order and fatigue strength for lithium disilicate.

One of the limitation of the present study is that the effect of the cementation procedures on the fatigue flexural strength of machined ceramic specimens, such as hydrofluoric acid etching and airborne sandblasting, was not evaluated. The effect of these variables in a CAD/CAM machined surface should be evaluated in future studies.

Finally, it is important to highlight that the polishing protocol adopted in the present study had just methodological purposes. This polishing protocol should not be extrapolated to the clinical practice, since the polishing of the intaglio surface of an all-ceramic restoration, with a reduction of 80 μm thickness, would increase cement thickness and result in fit problems. Actually, the present study points out the need of improving machining systems, developing milling protocols that induce less damage to the ceramic surface. Other machining variables, such as grit size and feed rate should be investigated to understand their effect on the machined surface.

5 Conclusions

CAD/CAM machining affected negatively the biaxial flexural fatigue strength of ceramics with different microstructures. The greatest reduction on the fatigue strength due to machining was observed in Y-TZP (40%), followed by lithium disilicate (33%) and leucite glass ceramic (29%), suggesting that soft machining may be as deleterious for the material's fatigue strength as hard machining. Higher values of roughness may be expected after machining.

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Table 1 – Ceramics used in the study with manufacturers data for chemical and physical characteristics.

Ceramic	Brand name; Manufacturer	Chemical composition	Vickers hardness (GPa)	Fracture toughness (MPa√m)	Machining	
					Set of bur	Manufacturer
Leucite-based glass ceramic	IPS Empress CAD; IvoclarVivadent	SiO_2 60-65 wt% Al_2O_3 16-20 wt% K_2O 10-14 wt% Na_2O 3.5-6.5 wt% other oxides 0.5-7 wt% pigments 0.2-1 wt%	6.2 fully sintered	1.3	Step bur 12 S (left side of the Cerec machine) Cylinder pointed bur 12 S (right side of the Cerec machine)	Sirona Dental Systems
Lithium disilicate-based glass ceramic	IPS e.max CAD; IvoclarVivadent	SiO_2 57-80 wt% Li_2O 11-19 wt% K_2O 0-13 wt% P_2O_5 0-11 wt% ZrO_2 0-8 wt% ZnO 0-8 wt% other and colouring oxides 0-12 wt%	5.4 partially crystallized 5.8 fully crystallized	2.0 - 2.5	Step bur 12 S (left side of the Cerec machine) Cylinder pointed bur 12 S (right side of the Cerec machine)	Sirona Dental Systems
Yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP)	Vita In-Ceram 2000 YZ for inLab; Vita Zahnfabrik	ZrO_2 91-94 wt% Y_2O_3 4-6 wt% HfO_2 2-4 wt% Al_2O_3< 0.1 wt% SiO_2< 0.1 wt% Na_2O < 0.1 wt%	11.77 fully sintered	5.9	Step bur 20 (left side of the Cerec machine) Cylinder pointed bur 20 (right side of the Cerec machine)	Sirona Dental Systems

Table 2 - Mean and standard deviation (MPa) of the monotonic and of the flexural fatigue strength; Weibull analysis of the fatigue data (characteristic flexural fatigue strength – σ_0; Weibull modulus – m; 95% confidence intervals); and roughness values (Ra and Rz) for the ceramics investigated, in both conditions: M – machining; MP – machining followed by polishing.

Ceramic	Surface condition	Monotonic flexural strength Mean (SD) (MPa)	Fatigue flexural strength Mean (SD) (MPa)	Weibull analysis of the fatigue data		Roughness	
				Characteristic fatigue strength [95% CI] (MPa)	Weibull modulus [95% CI]	Ra Mean (SD) (µm)	Rz Mean (SD) (µm)
Leucite	M	122.12 (14.11)*	69.50 (7.15)	72.61 [A] [69.28 – 76.04]	11.71 [A] [7.41 - 15.76]	1.59 [C] (0.17)	9.83 [C] (0.90)
	MP	166.48 (25.81)*	96.02 (14.72)	102.55 [B] [95.00 – 110.56]	7.18 [A] [4.48 - 9.71]	0.08 [D] (0.03)	0.51 [D] (0.17)
Lithium disilicate	M	269.52 (26.27)	180.71 (15.61)	187.71 [A] [180.30 - 195.30]	13.62 [A] [8.63 - 18.34]	1.84 [C] (0.18)	11.07 [C] (1.00)
	MP	479.39 (58.88)	267.10 (26.87)	278.93 [B] [266.04 - 292.22]	11.61 [A] [7.35 - 15.62]	0.13 [D] (0.06)	0.73 [D] (0.32)
Y-TZP	M	596.59 (57.18)	505.39 (75.88)	536.48 [A] [500.94 - 573.89]	8.01 [A] [5.07 - 10.78]	1.79 [C] (0.16)	11.21 [C] (1.07)
	MP	954.71 (104.29)	843.42 (117.00)	894.50 [B] [836.52 - 955.45]	8.19 [A] [5.19 - 11.03]	0.18 [D] (0.03)	1.71 [D] (0.37)

**results taken from Fraga et al. (2015)*

Different letters indicate statistically significant differences between M and MP conditions for each ceramic material (95% CI fail in overlapping for σ_0 and m; $p<0.05$ in Mann-Whitney test for Ra and Rz).

Table 3 – Mean and standard deviation (SD) of roughness (Ra) and flexural fatigue strength for each set of burs used in the machining of leucite, lithium disilicate and Y-TZP M groups. Spearman correlation coefficient (r_s) between machining order and Ra; and between machining order and flexural fatigue strength.

Ceramic	**Bur**	**Ra**			**Fatigue strength**		
		Mean (SD) - µm	***p-Value***	**r_s** ***p-Value***	**Mean (SD) - MPa**	***p-Value***	**r_s** ***p-Value***
Leucite	Set of Bur 1 n = 10	1.73 (0.86)	*0.000*	-0.88 *0.001*	69.91 (7.35)	*0.241*	0.74 *0.013*
	Set of Bur 2 n = 10	1.45 (0.81)		-0.21 *0.556*	73.88 (7.25)		0.44 *0.200*
Lithium disilicate	Set of Bur 1 n = 9	1.78 (0.18)	*0.450*	0.32 *0.406*	179.36 (13.16)	*0.114*	-0.07 *0.865*
	Set of Bur 2 n = 11	1.84 (0.18)		0.91 *0.000*	190.82 (16.88)		-0.21 0.537
Y-TZP	Set of Bur 1 n = 9	1.67 (0.10)	*0.001*	-0.63 *0.067*	554.25 (60.29)	*0.002*	0.73 *0.025*
	Set of Bur 2 n = 11	1.90 (0.15)		-0.47 *0.142*	459.31 (58.52)		0.50 *0.117*

* p<0.05 indicates significant difference.

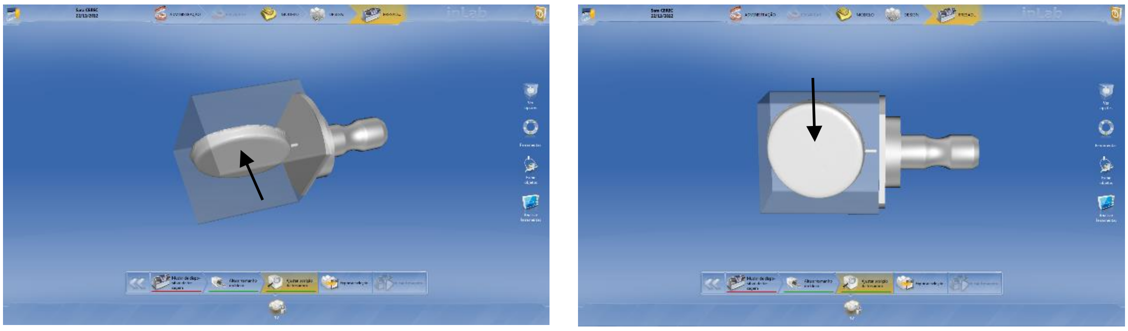
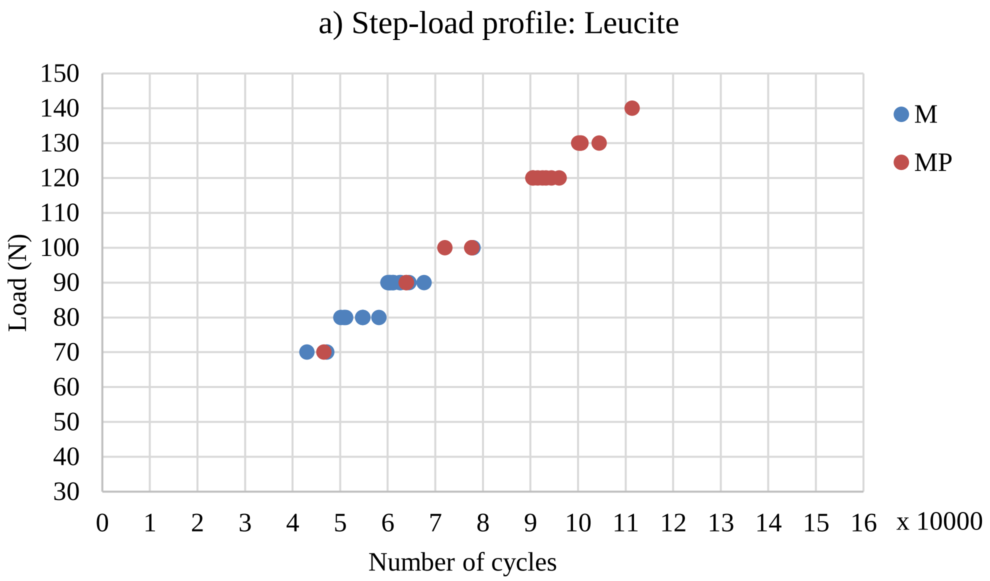
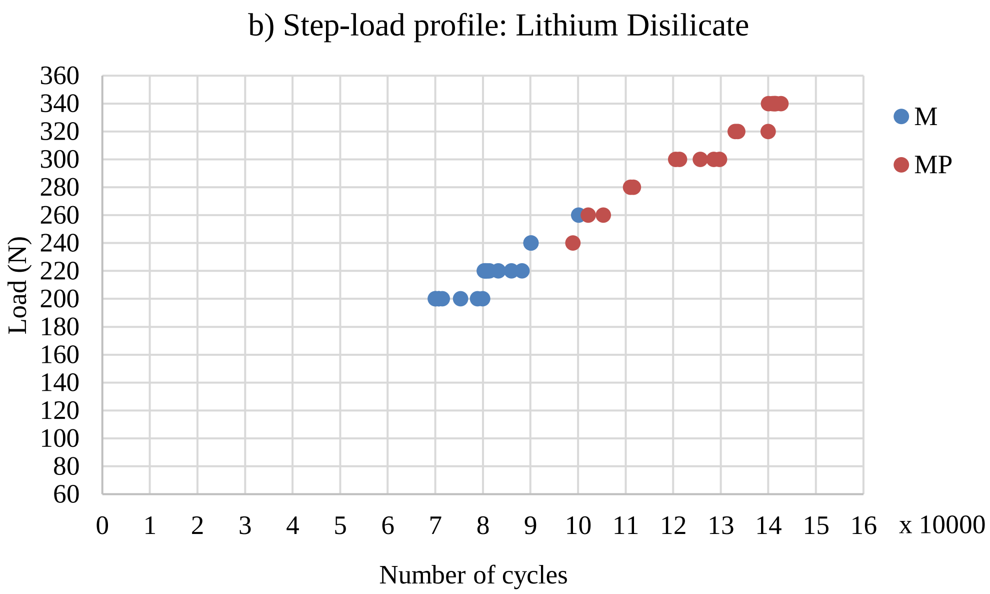


Fig. 1 – CEREC inLab 3D® software interface (version 4.0) showing a disc-shaped specimen, inside the ceramic block, ready for machining. The bottom surface of the disc (indicated by the black arrow) corresponded to the inner surface of a machined crown.


a) Step-load profile: Leucite
Load (N)
150
140
130
120
110
100
90
80
70
60
50
40
30
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 x 10000
Number of cycles
M
MP


b) Step-load profile: Lithium Disilicate
Load (N)
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 x 10000
Number of cycles
M
MP

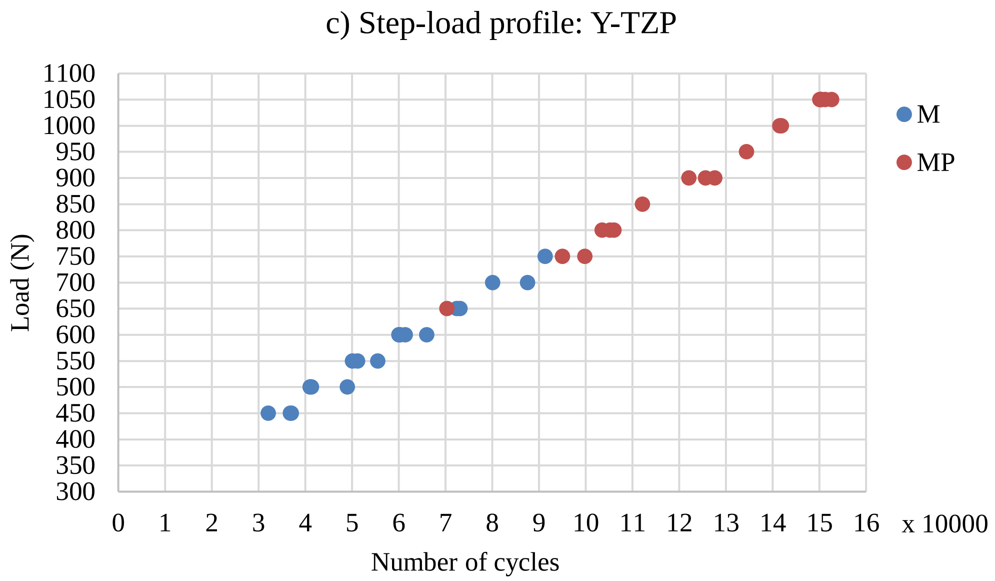
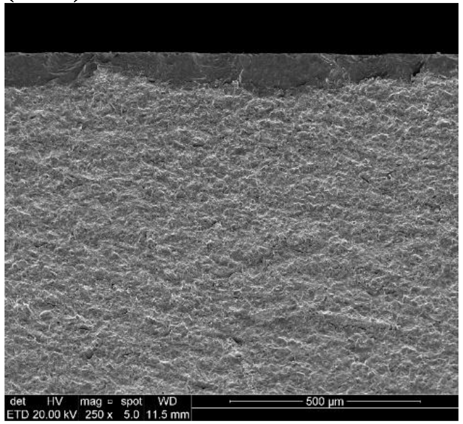
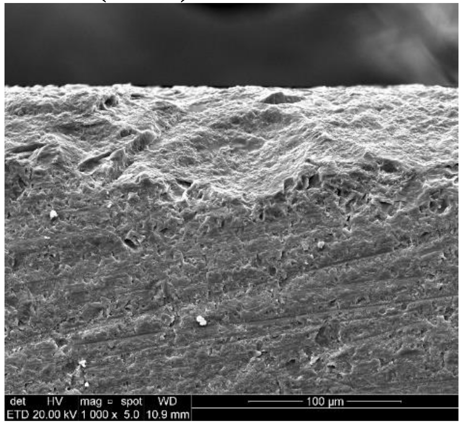


Fig. 2 – Step-load profile of leucite (a), lithium disilicate (b) and Y-TZP groups (c). The blue circles represent the fractured specimens of the M groups; the red circles represent the fractured specimens of the MP groups.

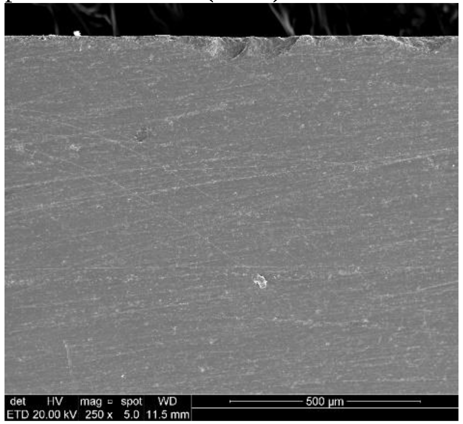
a1) Leucite – M – machined surface (250x)



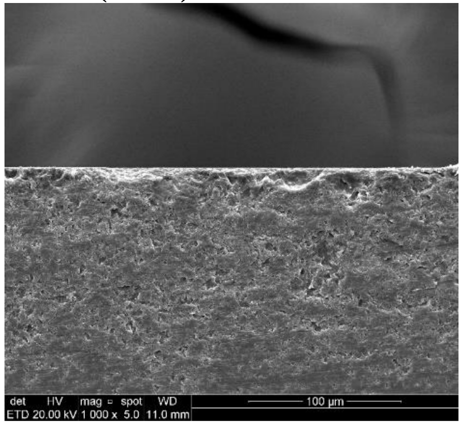
a2) Leucite – M – cross sectional surface (1000x)



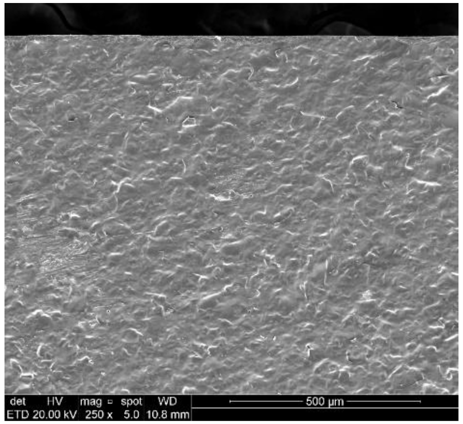
a3) Leucite – MP – machined and polished surface (250x)



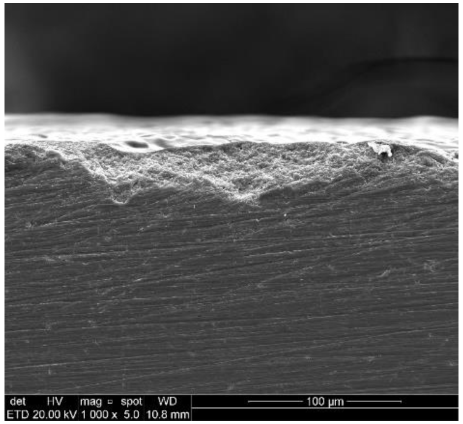
a4) Leucite – MP – cross sectional surface (1000x)



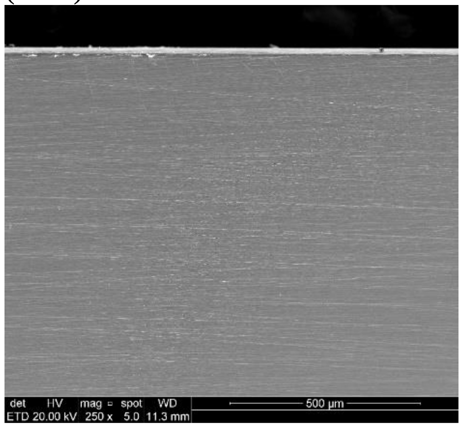
b1) Lithium disilicate – M – machined surface (250x)



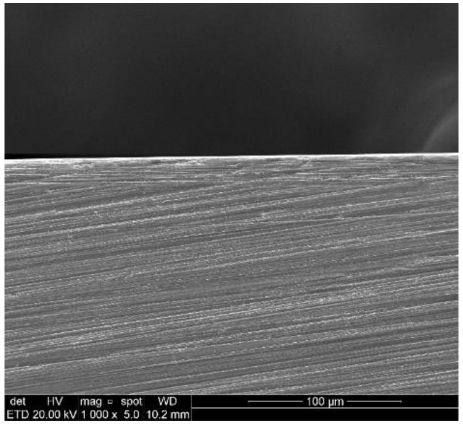
b2) Lithium disilicate – M – cross sectional surface (1000x)



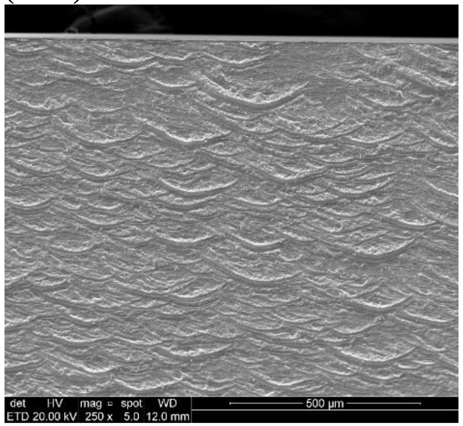
b3) Lithium disilicate – MP – machined and polished surface (250x)



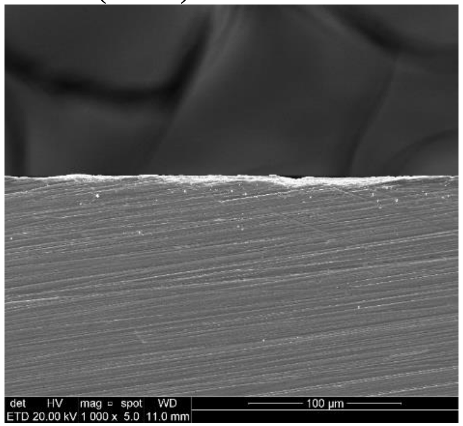
b4) Lithium disilicate – MP – cross sectional surface (1000x)



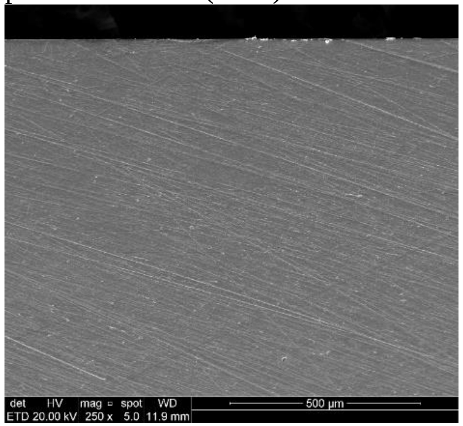
c1) Y-TZP – M – machined surface (250x)



c2) Y-TZP – M – cross sectional surface (1000x)



c3) Y-TZP – MP – machined and polished surface (250x)



c4) Y-TZP – MP – cross sectional surface (1000x)

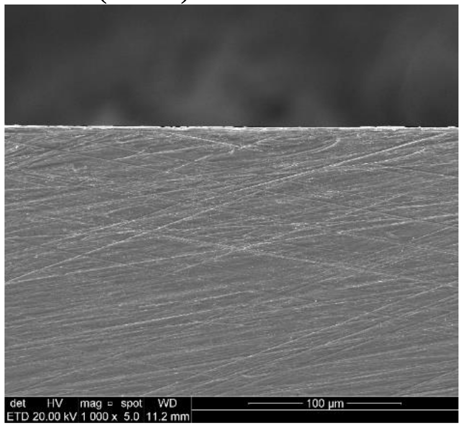


Fig. 3 – FESEM images of the M and MP surface for each ceramic material. It is possible to notice that M resulted in a rough surface, with the introduction of surface defects. Polishing seemed effective in removing these defects.

4 ARTIGO 2 – *LOADING FREQUENCIES UP TO 20 HZ AS AN ALTERNATIVE TO ACCELERATE FATIGUE STRENGTH TESTS IN A Y-TZP CERAMIC*

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Loading frequencies up to 20 Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic

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ABSTRACT

Considering the interest of the research community in the fatigue behavior of all-ceramic restorations and the time consumed in low-frequency cyclic fatigue tests, this study aimed to investigate the influence of the loading frequency on the zirconia fatigue strength. The biaxial flexural fatigue strength of Y-TZP discs was determined by the staircase approach after 500,000 cycles. The investigated frequencies were 2 Hz (control - simulation of the chewing activity; n = 20), 10 Hz (n = 20), 20 Hz (n = 20), and 40 Hz (n = 21). The fatigue strength data were analyzed using one-way ANOVA and *post hoc* Tukey`s test ($\alpha = 0.05$). Pearson coefficient (r) was calculated to assess the existence of a correlation between fatigue strength and loading frequency. X-ray diffraction analysis was used to determine the relative amount of monoclinic phase under each fatigue test condition. The fatigue strength was significantly higher for 40 Hz group (630.7 ± 62.1 MPa) and did not differ among the groups 2 Hz (550.3 ± 89.7 MPa), 10 Hz (574.0 ± 47 MPa) and 20 Hz (605.1 ± 30.7 MPa). Pearson correlation coefficient indicated a significantly moderate correlation (r = 0.57) between fatigue strength and loading frequency. The percentage of monoclinic phase was similar among the groups. Therefore, the use of loading frequencies up to 20 Hz seems a good alternative to expedite the cycling strength fatigue tests in polycrystalline ceramics without significantly changing the fatigue behavior showed by zirconia in tests employing the frequency of the masticatory cycle.

Keywords: fatigue; mechanical cycling; load frequency; zirconium oxide partially stabilized by yttrium.

1 INTRODUCTION

Despite the advances in dental materials science, including the introduction of Computer-Aided Design/Computer-Aided Machining (CAD/CAM) systems and the development of high-strength ceramics such as zirconia, dental ceramics still exhibit a brittle nature. As a result, these materials support little or no plastic deformation and suffer catastrophic failures, while their strength is sensitive to the presence of defects (Kelly, 1995, 2004).

In the oral environment, ceramic restorations are susceptible to fatigue failure, mainly due to the presence of cyclic masticatory forces and moisture (Zhang et al., 2013). Fatigue failure may be defined as the fracture of the material due to progressive brittle cracking under repeated cyclic stresses with intensity below the material normal strength (Wiskott et al., 1995).

Owing to high costs and ethical considerations, many issues related to restorative dental materials are difficult to evaluate in clinical studies (Kelly, 2006). Laboratory fatigue tests involving the application of a cyclic loading have been employed to partly reproduce the clinical conditions to which restorative materials are subjected during function. Analyses of *in vitro* fatigue experiments allow inferring on the survival probability of ceramic restorations (Lohbauer et al., 2008). Considering the high costs involved in the replacement of all-ceramic failed restorations, the evaluation of the ceramic fatigue strength in laboratory tests is strongly desired by clinicians and researchers, contributing thus to increase the number of studies on this subject (Baran et al., 2001).

Cyclic fatigue tests require the determination of some variables such as the loading frequency (number of loading cycles per second). Frequencies between 1 Hz and 2 Hz have been employed in many cyclic fatigue studies, considering that the chewing activity mainly occurs in the range of 0.94–2.17 Hz (Po et al., 2011). However, at low frequencies, the data collection becomes very time consuming. According to Wiskott et al. (1995), fatigue tests should be conducted with at least 10^6 cycles to provide clinical relevance. This number of cycles would be equivalent to one year of function.

Performing a cyclic fatigue test of 10^6 cycles at a frequency of 2 Hz (two load cycles per second) with a single station fatigue machine, approximately six days would be necessary to conclude the mechanical cycling for one specimen. At a higher frequency of 20 Hz, this time would be reduced to 14 h, clearly optimizing the data collection.

The effect of the load frequency on the cyclic fatigue behavior of ceramic materials is still not completely understood. Kelly et al. (2010) reported a slight increase in the cyclic fatigue load of a glass-infiltrated alumina when a frequency of 20 Hz was used (602.5 N), compared with the results obtained at 2 Hz (569.64 N). In the study of Joshi et al. (2014), no difference in terms of characteristic lifetimes was found for a glass-ceramic fluorapatite when the cyclic test was performed at frequencies of 2 Hz (10,800 seconds) and 10 Hz (11,600 seconds).

The excellent mechanical properties of yttria-stabilized polycrystalline tetragonal zirconia (Y-TZP) ceramics can be attributed to the transformation toughening mechanism that acts to resist crack propagation. Under stress, the Y-TZP tetragonal phase may transform into monoclinic state, resulting in 3-4% volume expansion, which induces compressive stress in the area of the crack, hindering further crack propagation. However, this phenomenon can also alter the phase integrity of the material, increasing its susceptibility to aging (Denry and Kelly, 2008). Mechanical and thermo-mechanical cycling are some of the methods that have been employed to promote low temperature degradation (LTD) in zirconia (Cotes et al., 2014), a phenomenon that is exacerbated by the presence of water (Chevalier et al., 2007). It is important to highlight that there is no data on literature regarding the influence of load frequency on cyclic fatigue behavior of Y-TZP ceramics.

Therefore, considering the increasing interest in the fatigue behavior of restorative materials, the extensive time consumed in low-frequency cyclic fatigue tests, and the lack of knowledge about the effect of the load frequency on the ceramic fatigue strength, this study aimed to investigate the influence of the frequency (2 Hz, control; 10 Hz; 20 Hz; 40 Hz) on the fatigue strength of a Y-TZP ceramic. The hypothesis was that the test frequency up to 40 Hz would not affect the fatigue strength of the Y-TZP ceramic at a lifetime of 500,000 cycles.

2 MATERIALS AND METHODS

2.1 Specimen preparation

Presintered zirconia blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Liechtenstein, lot nº RX0008) were ground into cylinders in a polishing machine (EcoMet/AutoMet 250, Buehler, United States) using a 400 grit silicon carbide paper under water refrigeration. The cylinders were then cut with a diamond saw (Labcut 1010, Extec Corporation, United States) under water cooling to obtain disc shaped Y-TZP specimens with initial dimensions of 18 mm in diameter and 1.7 mm in thickness (Fig. 1). In order to standardize the surface condition and to remove the irregularities introduced by cutting, the discs were hand-polished under water cooling, with a 1200 grit silicon carbide paper until the thickness of 1.5 mm. Then, the samples were cleaned in an isopropyl alcohol ultrasonic bath for 10 min, and sintered in a Vita Zyrcomat (Vita Zahnfabrik, Germany) furnace at a temperature of 1530 °C for 120 min. The final dimensions of the discs were 14.5 mm in diameter and 1.21 ± 0.03 mm in thickness.

After sintering, the discs were randomly divided into four groups according to the frequency applied in the fatigue test (2, 10, 20, and 40 Hz). Three additional discs were employed in a monotonic test.

The aim of the polishing protocol and of the randomization procedure was to ensure that the surface flaw population would be equally distributed among the groups.

2.2 Monotonic biaxial flexural strength

Prior to fatigue testing, the monotonic biaxial flexural strength was determined from three Y-TZP discs, using a piston-on-three ball assembly, according to ISO 6872:2008, in a universal testing machine (DL-1000 Emic, Brazil). The disc was positioned on the top of three steel spheres (2.5 mm in diameter, 120^o apart and forming a circle of 10 mm diameter), with a load applied at a rate of 1 mm/min, perpendicular to the center of the top surface of the disc, by a circular cylinder steel piston with a 1.4 mm diameter flat tip (Fig. 2). The monotonic fracture strength (σ_m), in MPa, was calculated using Eqs. (1), (2) and (3) (ISO 6872, 2008):

$$\sigma_m = -0.2387P(X - Y)/b^2 \qquad \text{Eq. (1)}$$

$$X = (1 + \nu)\ln(B/C)^2 + [(1 - \nu)/2](B/C)^2 \qquad \text{Eq. (2)}$$

$$Y = (1 + \nu)\left[1 + \ln A/C^2\right] + (1 - \nu)(A/C)^2 \qquad \text{Eq. (3)}$$

where P is the load at fracture (N), b is the disc thickness (mm), ν is Poisson's ratio (0.25), A is the support ball radius (5 mm), B is the radius of the tip of the piston (0.7 mm), and C is the specimen radius (7.25 mm).

2.3 Biaxial flexural fatigue strength

The fatigue test was conducted in an electric machine (Instron ElectroPuls E3000, Instron Corporation, United States) using the same piston-on-three ball configuration (ISO 6872, 2008) in water.

The flexural fatigue strength was determined for 500,000 cycles, using the staircase approach method described by Collins (1993). Sinusoidal loading was applied with amplitude ranging from a minimum of 10 MPa to the maximum tensile stress. Four different frequencies were tested: 2 Hz (control group), 10 Hz, 20 Hz, and 40 Hz.

To perform the staircase test, the number of cycles is previously set. The first specimen is tested at a stress lower than the maximum stress supported by the material in a corresponding static test. If the specimen fails before reaching the desired lifespan, the stress level is decreased by a preselected increment and the second specimen is tested at a new lower stress level. If the first specimen reaches the desired lifespan, the stress level is increased by the preselected increment and the second specimen is tested at this new higher stress level. The test is continued in this manner, with each succeeding specimen being tested at a stress level that is one increment above or below its predecessor, depending on whether the predecessor succeeded or failed.

Based on the results of the monotonic test (n = 3; mean monotonic biaxial flexural strength = 758.53 MPa; standard deviation = 134.67 MPa), the step size adopted was 38 MPa (equivalent to 10% of 0.5 σ_m). The first specimen of each group was tested with a maximum tensile stress of 455.26 MPa (equivalent to 0.5 σ_m plus two step sizes). If the specimen failed before reaching the life of interest (500,000 cycles), the stress level was decreased by one step size (38 MPa) and the next specimen was tested at this new lower stress level. If the specimen survived the 500,000 cycles, the stress level was increased by one step size (38 MPa) and the next specimen was tested at this new higher stress level. The test was continued in this manner in sequence, with each succeeding specimen being tested at a stress level that was one increment above or below its predecessor.

The sample size was 20 discs per group, except for the 40 Hz group, in which 21 discs were tested to provide at least 15 specimens after the beginning of the up-and-down character (point where the first reversal occurs) (Collins, 1993).

The load required to achieve the desired tensile stress was calculated from Eqs. (1), (2) and (3) (ISO 6872, 2008), considering the thickness of each disc. Thus, all fatigue tests were controlled by tension.

The mean fatigue flexural strength (σ_f) and the standard deviation (s) were calculated based on the data of the least frequent event (survival or failure), using the method described by Collins, 1993 (Eqs. 4 and 5):

$$\sigma_f = \sigma_{f0} + d[{}^{\sum in_i}/_{\sum n_i} \pm {}^{1}/_{2}] \quad \text{Eq. (4)}$$

$$s = 1{,}62d\ \{[(\sum n_i \sum i^2 n_i - (\sum in_i)^2)/(\sum n_i)^2] + 0{,}029\} \quad \text{Eq. (5)}$$

If: $[(\sum n_i \sum i^2 n_i - (\sum in_i)^2)/(\sum n_i)^2] \geq 0.3$

where σ_{f0} is the lowest stress level considered in the analysis and d is the step size. In Eq. (4), the negative sign is used if the least frequent event is failure; otherwise the positive sign is used. The lowest stress level considered is designated as i = 0, the next level as i = 1, etc.; n_i is the number of failures or survivals at the given stress level.

The test machine was adjusted to stop running (by displacement control) every time the specimen failed. Then, the number of cycles until fracture was registered.

2.4 Phase analysis by x-ray diffraction (XRD)

Quantitative analysis of phase transformation was conducted (n = 2) to determine the relative amount of monoclinic phase and depth of the transformed layer under each fatigue test condition (2 Hz, control; 10 Hz; 20 Hz; 40 Hz), using an x-ray diffractometer (D8 Advanced XRD, Bruker AXS GmbH, Germany) at a wavelength of 1.5416 Å (Cu K_α); the diffraction data were collected within a 2θ range of 25°-35° at a step interval of 1s and step size of 0.03°.

The amount (X_m) and the volumetric fraction (F_m) of the monoclinic phase were calculated based on the method introduced by Garvie and Nicholson (1972), modified by Toraya et al. (1984) (Eqs. 6 and 7):

$$X_m = \frac{(-111)_M + (111)_M}{(-111)_M + (111)_M + (101)_T} \quad \text{Eq. (6)}$$

$$F_m = \frac{1.311 X_m}{1 + 0.311 X_m} \quad \text{Eq. (7)}$$

where $(-111)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ = 28° and 2θ = 31.2°, respectively) and $(101)_T$ indicates the intensity of the respective tetragonal peak (2θ = 30°).

The depth of the transformed layer (PZT) was calculated based on the amount of monoclinic phase, considering that a constant fraction of grains had symmetrically transformed to the monoclinic phase along the surface, as described by Kosmac et al. (1981) (Eq. 8):

$$PZT = \left(\frac{sen\theta}{2\mu}\right)\left[ln\left(\frac{1}{1-F_m}\right)\right] \quad \text{Eq. (8)}$$

where θ = 15° (angle of reflection), μ = 0.0642 is the absorption coefficient, and F_m is the amount of monoclinic phase obtained by Eqs. (6) and (7).

2.5 Fractographic analysis

After the mechanical tests, the specimens were analyzed in a light microscope (Stereo Discovery V20; Carl Zeiss, Gottingen, Germany) and then representative discs from each evaluated condition were submitted to scanning electron microscopy (SEM) aiming to identify the surface where the fracture initiate.

2.6 Statistical analysis

Fatigue strength values were analyzed using one-way ANOVA and *post hoc* Tukey`s test ($\alpha = 0.05$), since the data presented homogeneity of variances ($p > 0.05$ based on the Levene test) and normal distribution ($p > 0.05$ based on the Shapiro-Wilk test). Pearson coefficient (r) was calculated to assess the existence of a correlation between fatigue strength and load frequency.

3 RESULTS

The biaxial fatigue strength was significantly higher for 40 Hz (630.7 ± 62.1 MPa, $p \leq 0.026$) and did not differ among the groups 2 Hz (550.3 ± 89.7 MPa), 10 Hz (574.0 ± 47 MPa) and 20 Hz (605.1 ± 30.7 MPa) ($p \geq 0.823$) (Table 1). Pearson correlation coefficient demonstrated a significantly ($p = 0.001$) moderate correlation ($r = 0.57$) between fatigue strength and load frequency (Crespo, 1997). The percentage of monoclinic phase and the depth of the transformed layer were similar among the groups (Table 1).

The pattern of runouts and failures for each group is described in Fig. 3.

The number of cycles until fracture ranged from 170 to 42,583 at 2 Hz, from 69 to 34,319 at 10 Hz, from 654 to 84,546 at 20 Hz, and from 202 to 35,383 at 40 Hz.

In all specimens, as expected, failures seem to initiate on the surface subjected to tensile stress during the flexural test. A similar failure pattern may be identified among the groups in the SEM images (Fig. 4).

4 DISCUSSION

The main scientific contribution of the present study is to support researchers in the selection of an adequate load frequency to optimize future cyclic fatigue strength tests, as the use of higher frequencies reduces considerably the time necessary to conclude the mechanical cycling. The hyphothesis that the frequency would not affect the fatigue strength of the Y-TZP ceramic at a lifetime of 500,000 cycles was partially rejected. The Y-TZP fatigue strength was significantly higher when a load frequency of 40 Hz was applied (630.7 ± 62.1), in comparison with 2 Hz (550.3 ± 89.7), 10 Hz (574.0 ± 47.0) and 20 Hz (605.1 ± 30.7). Therefore, these results suggest that the cyclic fatigue strength tests at a maximum lifetime of 500,000 cycles can be expedited by using frequencies up to 20 Hz without statistically changing the fatigue strength of Y-TZP, compared to the frequency of the masticatory cycle (2 Hz).

The microstructure of the material and its sensitivity to the environment seem to affect how the load frequency influences the material fatigue strength. Some engineering material studies have shown that an increase of loading frequency has practically no effect on the fatigue limit of metals in air (Nonaka et al., 2014). However, in corrosive media, increasing the loading frequency usually results in an increase of the fatigue limit. As corrosion is time dependent, slow cycling rates will cause greater life reductions than accelerated fatigue cycling (ASM, 2008).

Joshi et al. (2014) reported that cyclic fatigue did not have a significant effect on the subcritical crack growth parameters of fluorapatite glass-ceramic. Only stress corrosion had a significant effect on the ceramic lifetime. Cyclic loading tests were performed at frequencies of 2 Hz and 10 Hz. Similar characteristic lifetimes were found at both frequencies (10,800 seconds at 2 Hz and 11,600 seconds at 10 Hz), indicating that specimens tested at 10 Hz (10 cycles per second) survived for a higher number of cycles than the specimens tested at 2 Hz (2 cycles per second). Thus, for predominantly glassy ceramics, in lifetime studies, the use of higher frequencies did not reduce the testing time.

Kelly et al. (2010) investigated the influence of frequency load (2 Hz, 10 Hz and 20 Hz) in the mean failure loads of veneered glass-infiltrated alumina discs cemented to flat dentin analog bases. A slightly positive relationship ($r = 0.68$) between cycling frequency and failure loads was reported. A small difference (approximately 5%) was found in the fatigue strength at 20 Hz (602.5 N) and 2 Hz (569.64 N). These results are in agreement with the present study, in which a moderate positive correlation ($r = 0.57$) was found between fatigue strength and load frequency. However, the fatigue strength mean was not significantly different among the groups 2 Hz, 10 Hz and 20 Hz.

The significant reduction in the mechanical cycling time (69 h per specimen to load 500,000 cycles at 2 Hz; 7 h per specimen to load 500,000 cycles at 20 Hz) did not influence the zirconia fatigue strength, which may suggest that Y-TZP has a high resistance to stress corrosion, a time-dependent phenomenon. However, a detailed study about the influence of the stress corrosion and cyclic degradation on the zirconia slow crack growth (SCG) parameters is necessary to confirm this hypothesis.

Zirconia is usually retained in a tetragonal form at room temperature by the addiction of stabilizing oxides, such as Y_2O_3, CaO, MgO or CeO_2. The tetragonal crystallographic form is metastable. When the material is subjected to a stress, the tetragonal phase may transform into monoclinic state, resulting in 3-4% volume expansion which induces compressive stresses, thereby closing the crack tip and hindering further crack propagation (Denry and Kelly, 2008). Phase analysis of partially stabilized zirconia by x-ray diffraction (XRD) has permitted the quantitative determination of monoclinic contents present in the material (Paterson and Stevens, 1986). The relative amount of monoclinic phase and depth of the transformed layer occurred in a very similar way in all the investigated frequencies (Table 1).

One of the phenomenon involved in the cyclic fatigue strength of ceramic materials is the crack closure. This mechanism happens when the stress concentrated in the crack tip changes from tension to compression, during the cyclic loading. During crack closure can be observed attrition between crack walls, grain pull-out and grain crushing (which will create debris and enhance even more this mechanism), resulting in important microstructural changes and additional damage, reducing the material fatigue strength (Jian et al., 1993). The higher fatigue strength observed at the 40 Hz group may be explained as this increased frequency would not allow enough time to the occurrence of the crack closure mechanism, decreasing its effect on the Y-TZP fatigue strength.

Aiming to achieve a feasible testing time, the fatigue test was performed with a maximum of 500,000 cycles. The maximum number of cycles registered until fracture was 84,546 (20 Hz); thus, failures tended to occur at the beginning of the test, which indicate that 500,000 cycles may be considered suitable for the purpose of the present study. However, it is important to highlight that cyclic studies, aiming to simulate clinical conditions, should be performed with a higher number of cycles. According to Wiskott et al. (1997), one million cycles would represent one year in function. This number was reached assuming 3 periods of

15 minutes of chewing per day, at a chewing rate of 60 cycles per minute (1 Hz) (Wiskott et al., 1997).

The pattern of failure was similar for all groups, as illustrated in Fig. 4. The SEM images show some typical features of brittle material failures, such as the presence of hackles pointing to the failure origin, on the surface subjected to tensile stresses during the test (Quinn, 2007), indicating that the fractures did not occur due to possible high stresses concentration at the contact surface between the disc and the piston.

In the present study, a piston-on-three ball setup was used, in agreement with ISO 6872:2008, since biaxial strength testing of brittle materials have some advantages in comparison with uniaxial testing, such as: a simpler specimen preparation, as it did not require a chamfer; avoiding the effect of edge fracture, since the maximum tensile stress will be located at the center of the disc (Sadighpour et al., 2006).

Regarding the fatigue test, the staircase approach may be considered a useful testing method for determining the fatigue strength at any specified life (Collins, 1993). Fifteen to thirty specimens, after the beginning of the up-and-down character (point where the first reversal occurs), are required to adequately perform the test (Collins, 1993). Because of this, a sample size of 20 and 21 specimens was used in the present study.

Notably, the results reported in the present study are valid for cycling tests aiming to investigate the Y-TZP fatigue strength at a maximum lifetime of 500,000 cycles. For the extrapolation of these results and the indication of higher frequencies in general mechanical cycling tests, additional studies employing different fatigue methodologies, such as lifetime studies considering a longer time of the exposition of the material to the water, should be performed to better clarify the effect of the loading frequency on the fatigue behavior of Y-TZP.

5 CONCLUSIONS

There is a positive moderate correlation between fatigue strength and load frequency. However, the use of loading frequencies up to 20 Hz did not significantly affect the fatigue strength of Y-TZP at a lifetime of 500,000 cycles. Therefore, it appears to be a valid approach to accelerate the cyclic fatigue strength tests performed on this material at a maximum lifetime of 500,000 cycles.

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Table 1 – Fatigue strength, monoclinic phase content, depth of the transformed layer, and testing time per specimen for 500,000 mechanical cycles at different load frequencies (2 Hz, control; 10 Hz; 20 Hz; 40 Hz).

Load frequency	Time per specimen/ 500,000 cycles	Mean fatigue strength (standard deviation) - MPa	Monoclinic phase (standard deviation) - %	Depth of transformed layer (standard deviation) - µm
2 Hz (control)	69 h	550.3^{A} (89.7)	3.16 (0.11)	0.16 (0.006)
10 Hz	14 h	574.0^{A} (47.0)	3.76 (0.02)	0.19 (0.001)
20 Hz	7 h	605.1^{A} (30.7)	3.51 (0.33)	0.18 (0.017)
40 Hz	3 h	630.7^{B} (62.1)	4.66 (0.27)	0.24 (0.014)

Different letters indicate statistically significant differences ($p < 0.05$ in Tukey`s test)
**Monotonic biaxial flexural strength: 758.53 ± 134.67 MPa*

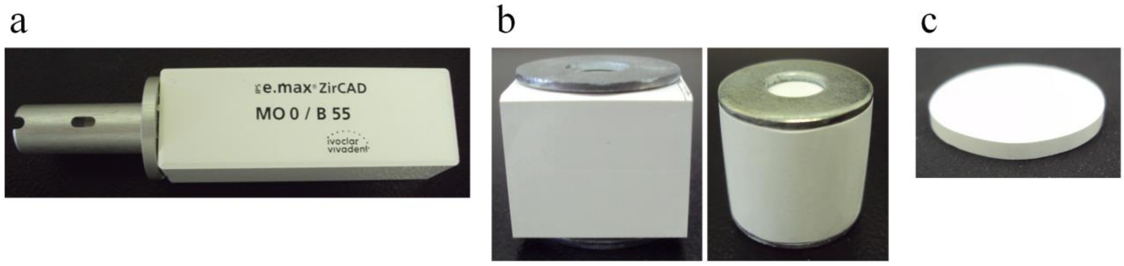


Fig. 1 – Specimen preparation: pre-sintered Y-TZP block (a); Y-TZP cylinder prepared by grinding with a 400 grit silicon carbide paper under water refrigeration in a polishing machine (b); pre-sintered Y-TZP disc (c). After that, the discs were polished and, then, sintered.

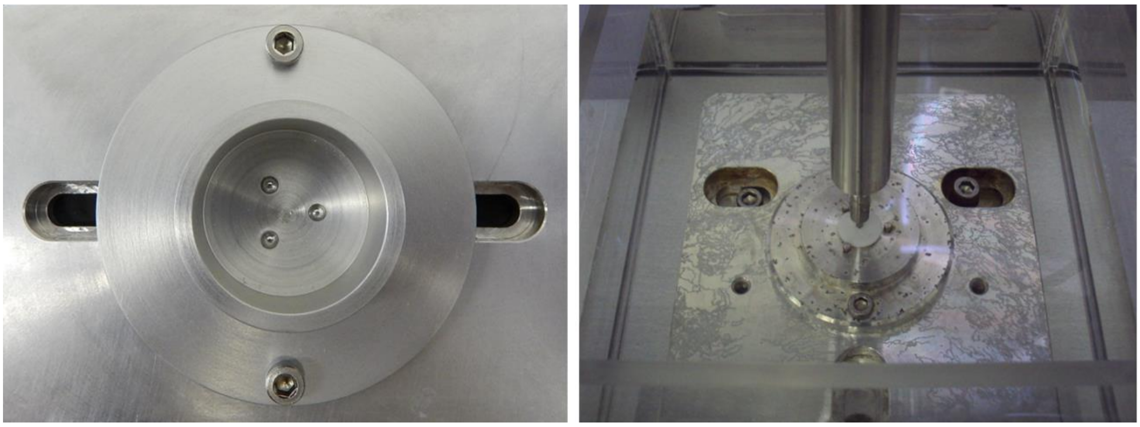
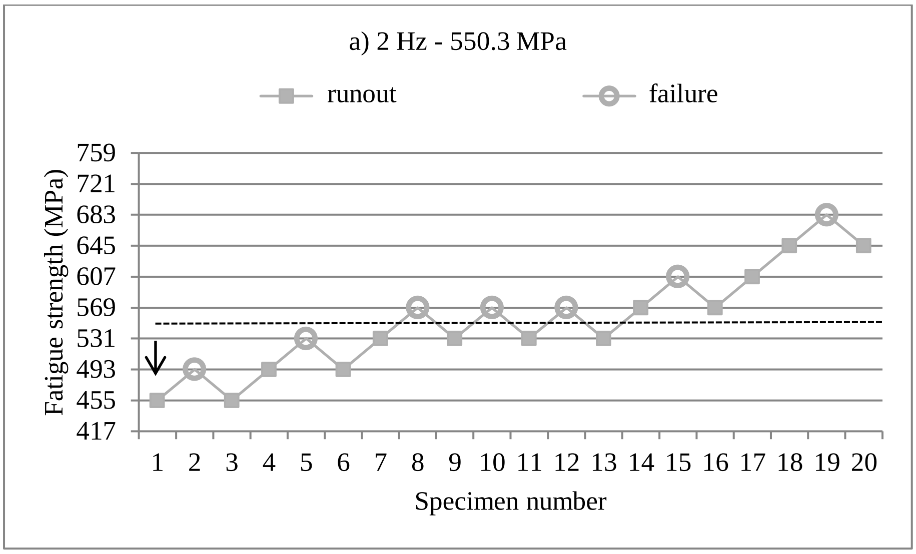
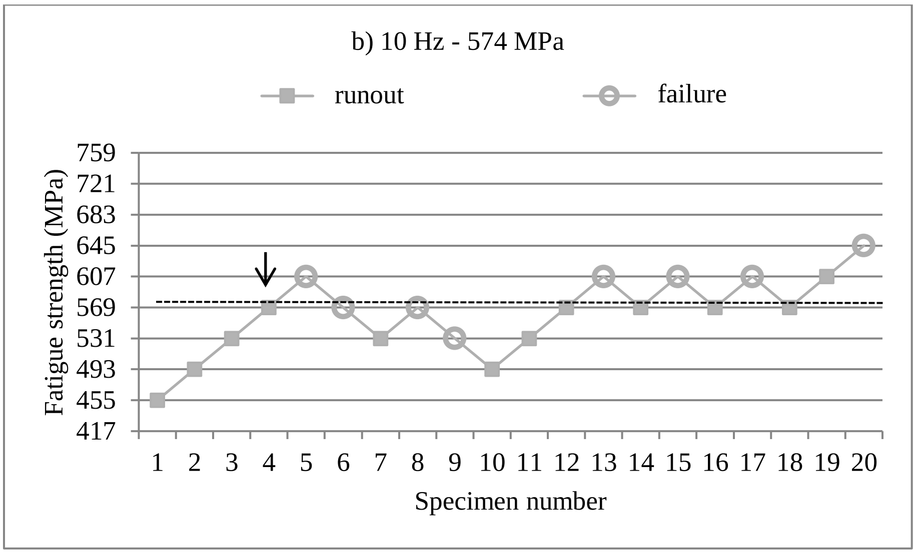
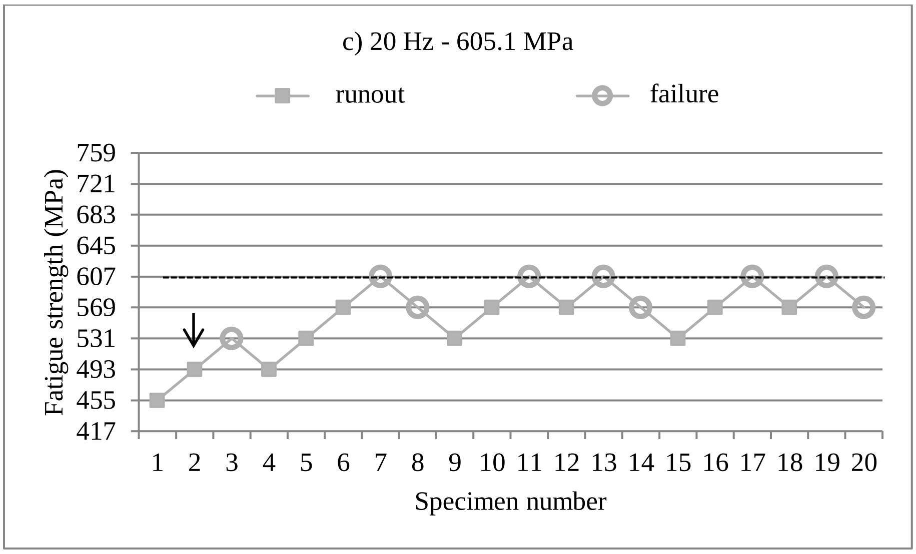


Fig. 2 – Piston-on-three ball device, according to ISO 6872:2008. The disc was positioned on the top of three steel spheres (2.5 mm in diameter, 120° apart and forming a circle of 10 mm diameter), with a load applied perpendicular to the center of the top surface of the disc, by a circular cylinder steel piston with a 1.4 mm diameter flat tip.


a) 2 Hz - 550.3 MPa
runout
failure
Fatigue strength (MPa)
759
721
683
645
607
569
531
493
455
417
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Specimen number


b) 10 Hz - 574 MPa
runout
failure
Fatigue strength (MPa)
759
721
683
645
607
569
531
493
455
417
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Specimen number


c) 20 Hz - 605.1 MPa
runout
failure
Fatigue strength (MPa)
759
721
683
645
607
569
531
493
455
417
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Specimen number

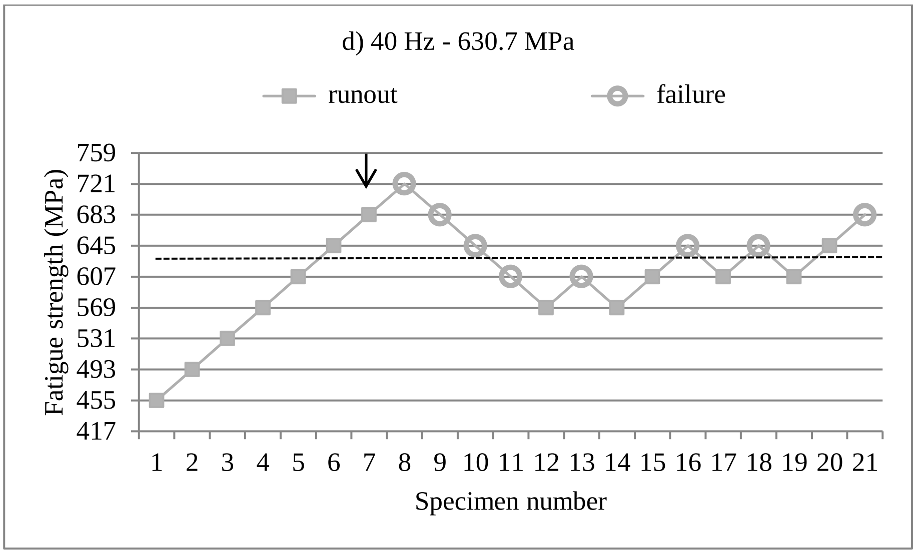
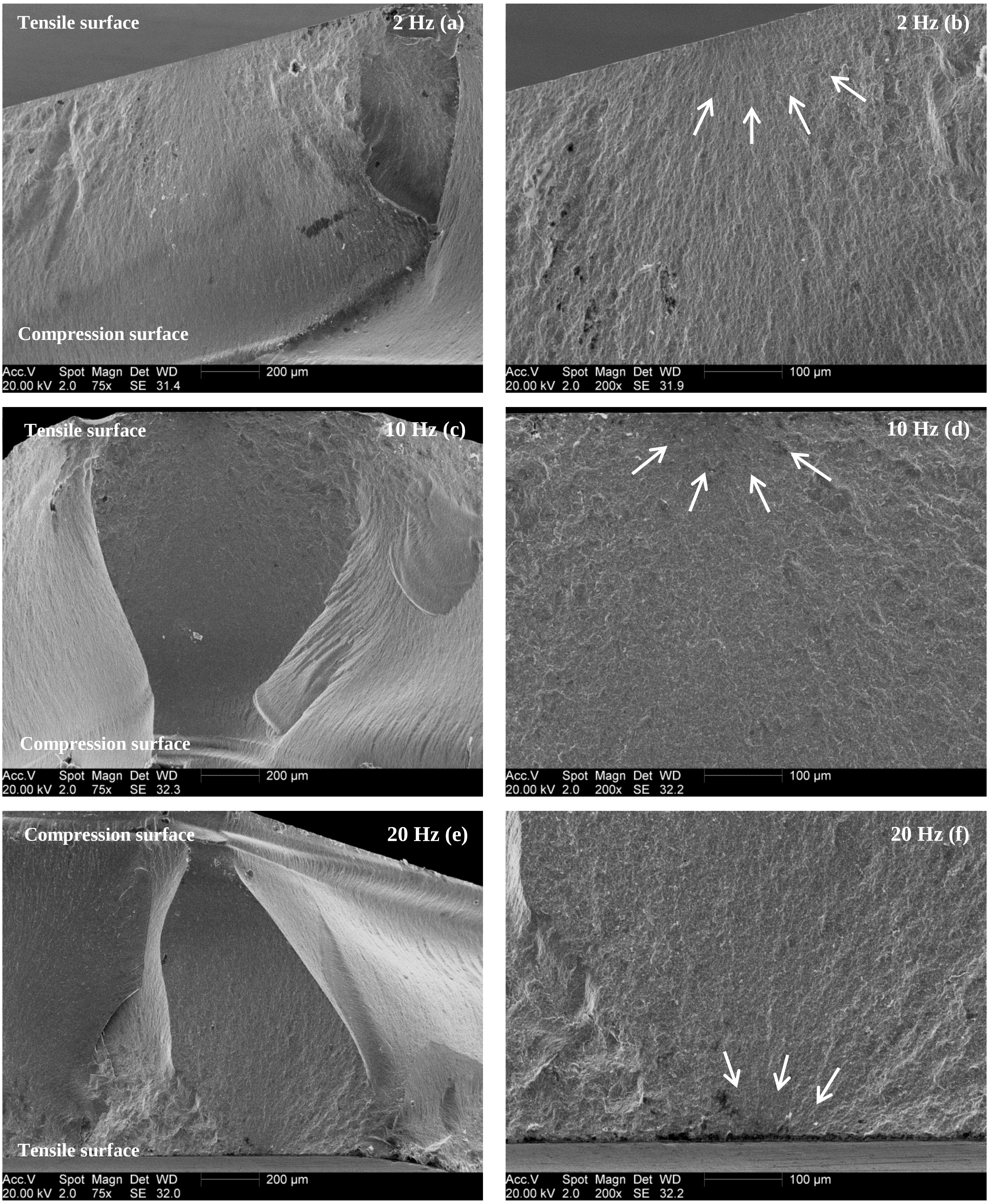


Fig. 3 – Staircase sensitivity test results during mechanical cycling (500,000 cycles) at 2 Hz (a), 10 Hz (b), 20 Hz (c), and 40 Hz (d). The arrows indicate the stress level at which the up-and-down character started. The dashed lines indicate the fatigue strength mean.


Tensile surface
2 Hz (a)
Compression surface
Acc.V Spot Magn Det WD 200 µm
20.00 kV 2.0 75x SE 31.4
2 Hz (b)
Acc.V Spot Magn Det WD 100 µm
20.00 kV 2.0 200x SE 31.9
Tensile surface
10 Hz (c)
Compression surface
Acc.V Spot Magn Det WD 200 µm
20.00 kV 2.0 75x SE 32.3
10 Hz (d)
Acc.V Spot Magn Det WD 100 µm
20.00 kV 2.0 200x SE 32.2
Compression surface
20 Hz (e)
Tensile surface
Acc.V Spot Magn Det WD 200 µm
20.00 kV 2.0 75x SE 32.0
20 Hz (f)
Acc.V Spot Magn Det WD 100 µm
20.00 kV 2.0 200x SE 32.2

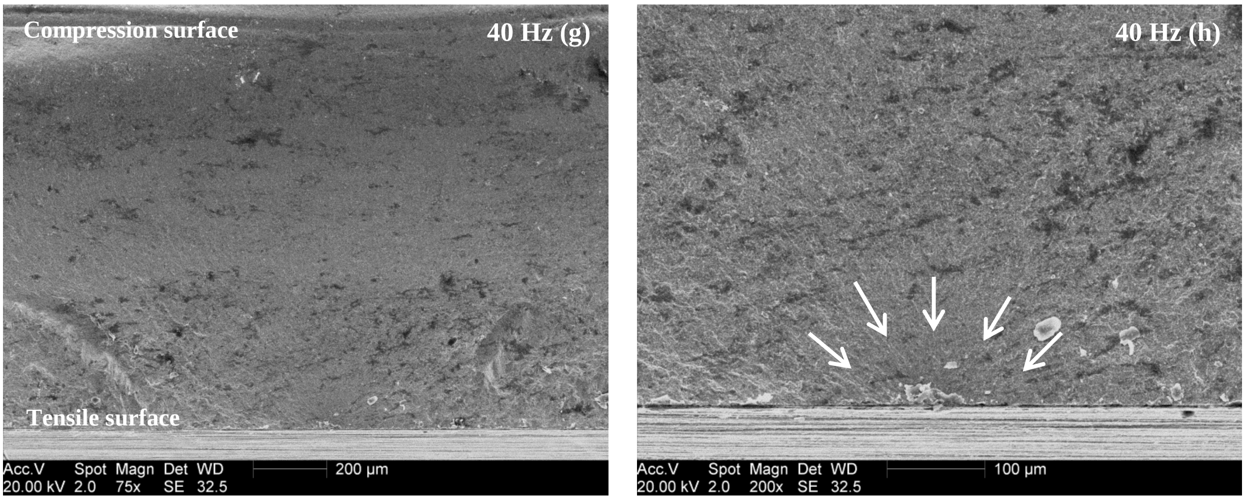


Fig. 4 – Scanning electron microscopy images of the fracture surface of the discs subjected to fatigue at 2 Hz (a, b), 10 Hz (c, d), 20 Hz (e, f), and 40 Hz (g, h). Hackles pointing to the failure origin on the tensile surface can be observed (white arrows).

5 DISCUSSÃO

O uso da tecnologia CAD/CAM na Odontologia Restauradora tem se tornado cada vez mais difundido, abrangendo desde a confecção de restaurações simples até a realização de trabalhos reabilitadores complexos. Nesses sistemas, cerâmicas policristalinas são usinadas no estágio parcialmente sinterizado, com dimensões superiores ao trabalho final, a fim de compensar a contração esperada após a sinterização. Blocos de cerâmicas vítreas são fresadas nas dimensões finais do trabalho restaurador, com precisão de micrômetros. Novos produtos, como câmeras intraorais para moldagem digital e materiais para usinagem, são regularmente lançados no mercado. Todos os fatos mencionados exemplificam o grande investimento e a alta tecnologia envolvida nos sistemas CAD/CAM (KELLY, 2006; LEBLON et al., 2016). Entretanto, limitações e necessidade de melhorias ainda se fazem presentes.

Os resultados apresentados no artigo ***"Impact of the CAD/CAM machining on the fatigue behavior of glass and polycrystalline ceramics"*** indicam que a usinagem é capaz de introduzir defeitos na superfície de cerâmicas com diferentes microestruturas, com efeito deletério na resistência à fadiga flexural. Esses achados são corroborados pela análise clínica de coroas que falharam clinicamente, em que irregularidades decorrentes da fresagem foram bastante nítidas e, em muitos casos, correspondiam ao ponto de origem da falha (ØILO; QUINN, 2016).

Diferenças em termos de rugosidade superficial e resistência à fadiga flexural encontradas para os pares de brocas usados na usinagem de um mesmo material cerâmico sugerem uma deficiência na padronização dos instrumentos diamantados de corte, com possível efeito negativo na previsibilidade clínica das restaurações totalmente cerâmicas.

Considerando-se o exposto, fica clara a necessidade de se buscar alternativas que resultem em menor introdução de defeitos superficiais durante o processo de usinagem de cerâmicas parcialmente e totalmente sinterizadas. Nesse contexto, o estudo do efeito de outras variáveis, a exemplo do tamanho das partículas de diamante presentes nas brocas e da velocidade de desgaste, é fundamental para o aprimoramento dos sistemas de usinagem.

Tendo-se em vista que as cerâmicas são materiais frágeis (KELLY, 1995), sujeitas ao fenômeno de crescimento lento e subcrítico de trincas (GONZAGA et al., 2011), é imperativo que o efeito de procedimentos capazes de introduzir defeitos superficiais seja investigado sob condições de fadiga. Em função disso, no primeiro estudo apresentado nessa tese, ensaio de fadiga cíclica foi empregado para avaliar o impacto da usinagem na resistência de cerâmicas com diferentes microestruturas.

Como comentado na revisão de literatura, ensaios cíclicos envolvem a determinação prévia de alguns parâmetros, como a frequência de aplicação de carga. O uso de frequências muito baixas pode tornar a coleta dos dados muito lenta e, até mesmo, inviabilizar a realização do estudo. Por outro lado, o uso de frequências muito altas pode produzir resultados equivocados, por não haver tempo hábil para o material reagir adequadamente à carga aplicada (ROSENTRITT; BEHR; PREIS, 2016).

Os resultados apresentados no artigo ***"Loading frequencies up to 20 Hz as an alternative to accelerate fatigue strength tests in a Y-TZP ceramic"*** mostraram que é possível acelerar os ensaios de resistência à fadiga cíclica em cerâmicas policristalinas em até 20 Hz, sem alteração dos resultados obtidos com frequência de 2 Hz. Tal estudo reveste-se de importância à medida que poderá ser usado para embasar a utilização de frequências superiores em ensaios de resistência à fadiga, além de viabilizar o desenvolvimento de trabalhos que avaliem o comportamento à fadiga em um maior número de ciclos.

Faz-se importante enfatizar que os resultados mencionados são válidos apenas para cerâmicas policristalinas, e não devem ser extrapolados para cerâmicas com conteúdo vítreo. As cerâmicas vítreas caracterizam-se por serem mais susceptíveis do fenômeno de SCG (BORBA et al., 2011), fazendo com que o meio úmido exerça um maior efeito na sua resistência. Portanto, a aceleração do ensaio cíclico, por meio do uso de frequências mais altas, poderia acarretar em valores de resistência superiores quando comparados aos obtidos em baixas frequências. Entretanto, tal hipótese só poderá ser avaliada por meio da condução de um ensaio in vitro adequadamente delineado.

Na presente tese, dois métodos de avaliação da resistência à fadiga cíclica foram apresentados: *staircase* e *step-test*. Ambos os ensaios permitem a obtenção de valores de média e de desvio padrão para a resistência à fadiga. Entretanto, algumas considerações devem ser feitas. No método *staircase*, apesar de ser simples e de natureza sequencial, os testes são normalmente conduzidos em uma faixa de cargas muito estreita, ou seja, valores extremos não são considerados, fato que poderia mascarar a performance do material sob condições mais extremas (BONFANTE; COELHO, 2016). No método *step-test*, o tempo de vida, para o qual o valor de resistência à fadiga será calculado, corresponderá ao número de ciclos empregado no *step*, e não ao somatório total de ciclos suportado pelo material. Na realização dos cálculos de resistência, assume-se que o dano acumulado e o número de ciclos empregados se relacionem de forma linear (NICHOLAS, 2006). A grande limitação desse ensaio reside no fato de que o efeito do dano acumulado em cada *step* é desconhecido (COLLINS, 1993). Dessa forma, é importante que as peculiaridades e limitações de cada metodologia sejam consideradas a fim de evitar equívocos na interpretação dos resultados obtidos.

6 CONCLUSÃO

Com base nas investigações científicas apresentadas nessa tese, conclui-se que:

- A usinagem CAD/CAM apresenta um significativo efeito deletério no comportamento mecânico de materiais cerâmicos, reduzindo a resistência à fadiga em cerca de 40% para a Y-TZP, 33% para o dissilicato de lítio e 29% para a cerâmica vítrea reforçada por leucita. Assim, usinagem de materiais parcialmente sinterizados mostra-se tão deletéria quanto a usinagem de materiais totalmente sinterizados. Alternativas devem ser buscadas no sentindo de reduzir o dano ocasionado pela usinagem. Para tanto, é essencial que o impacto de outras variáveis, a exemplo do tamanho e da qualidade da partícula de diamante presente nas brocas, bem como da velocidade de desgaste, seja investigado.
- A determinação da frequência de aplicação de carga é um ponto bastante crítico no delineamento de estudos de fadiga cíclica. A utilização de frequências de até 20 Hz (20 ciclos por segundo) representa uma alternativa para acelerar os ensaios de resistência à fadiga em cerâmicas policristalinas, otimizando a coleta dos dados sem alterar significativamente os resultados.

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

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.

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Introduction

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not obvious.

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"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.

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• 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
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[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.

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