EGYPTIAN DENTAL JOURNAL

Vol. 63, 823:832, January, 2017

I.S.S.N 0070-9484

Fixed Prosthodontics, Dental materials, Conservative Dentistry and Endodontics

www.eda-egypt.org • Codex : 190/1701

EFFECT OF REPRESSING AND GLAZING ON COLOR REPRODUCTION, TRANSLUCENCY AND SURFACE ROUGHNESS OF LITHIUM DISILICATE GLASS-CERAMICS

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ABSTRACT

Statement of problem: During heat-pressing of lithium dislilicate glass-ceramics, It is more economical to press several restorations from one ingot at the same time. However, this is often not possible and may result in a considerable amount of leftover material (The sprues and button). It has been reported that the leftover materials after heat-pressing are reused (repressed) in some dental laboratories. Sufficient knowledge about the consequences of such procedure is not available. The issue is thereby raised whether the leftover material should be discarded or reused.

Purpose. The aim of the present study was to evaluate the effect of repressing and glazing on the color reproduction, translucency and surface roughness of lithium dislilicate pressable glass-ceramics (IPS e.max Press). As well as to describe the microstructural features present in pressed, and repressed material using **Xray diffraction, EDAX and SEM**

Materials and Methods: Twenty IPS e.max press discs (Pressed and Repressed groups n=10) of 15mm diameter, 1 mm thickness, in shade A3 were fabricated using heat pressed technique. **Only** the button parts and not the sprues were used for repressing. Color reproduction and translucency were measured using the Vita Easyshade spectrophotometer. Surface roughness was measured using a 3D laser scanning microscope. Crystalline structure and microstructural features present in pressed, and repressed material were described using Xray diffraction, EDAX and scanning electron microscope.

Results: Two-way analysis of variance (ANOVA), followed by Tuckey's HSD test at a significance level of $p < 0.05$ was performed. Regarding Color reproduction, Translucency and Roughness, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups

Conclusions: Lithium disilicate glass-ceramic materials have reached their maximum crystallinity prior to repressing. It is important to produce a pore–free structure by only repressing the left over buttons and not the left over sprues. Repressing has no significant effect on Color, translucency and surface roughness and Glazing is crucial to increase color reproduction, translucency and decrease surface roughness of IPS e.max Press.

KEY WORDS : Lithium disilicate, Repressing, Colour reproduction, Translucency, Glazing, Roughness

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INTRODUCTION

The appearance of natural teeth is best mimicked by ceramic materials.[1] In recent years the popularity of all-ceramic dental restorations has increased due to their high esthetic qualities and metal-free structure. Significant developments in all-ceramic materials have created wonderful opportunities for the fabrication of lifelike restorations that provide reliable, long-term results [2] However, all-ceramic dental materials, are inherently fragile in tension, affected by microcracking, flaws, and defects that may be introduced during thermal treatment or fabrication procedures. The fabrication process precision, and skills of individual dental technicians, may affect the reliability and clinical performance of all-ceramic restorations. Mechanical properties such as strength and optical properties such as color and translucency, are the first parameters assessed to understand the clinical potential and limits of dental ceramics. [3]

Heat-pressing has become a common technique to produce glass-ceramic dental restorations. In addition to its simplicity, this technique promotes better crystalline dispersion within a glass matrix, less porosity, and better marginal adaptation compared to sintering technique. IPS Empress was the original heat-pressed glass ceramic and leucite (SiO_2, Al_2O_3, $4K_2O$) is the main crystalline phase in this system. IPS Empress 2 has lithium disilicate ($Li_2O.2SiO_2$) as its main crystalline phase and is 60% crystalline when processed [2]

IPS e.max Press material has now replaced IPS Empress 2; it has improved mechanical properties and has significantly higher translucency. The microstructure consists of 70% lithium disilicate crystals embedded in a glassy matrix. These crystals are circular in morphology and measure 3 to 6 mm in length. IPS e.max Press is supplied for heat pressing in 2 sizes, a small ingot that weighs 3.2 g or a larger ingot that weighs 6.1 g. These ingots are pressed into a mold by an Alumina plunger under pressure from a pneumatic press furnace. After pressing and cooling, the sprues are removed, along with the remaining material (button). The sprues and buttons should be discarded and a new ingot should be used for a new pressing.[4]

It is more economical to press several restorations from one ingot at the same time. However, this often is not possible and may result in a considerable amount of leftover material (The remaining sprues and button). It has been reported that these remaining materials are recycled in some dental laboratories; sufficient knowledge about the safety and consequences of such treatment is not available. The issue is thereby raised whether these buttons can be repressed and recycled or should be discarded. Concerns have also been expressed regarding the change in the microstructure and possible degradation of the mechanical and optical properties of these materials, as a result of multiple processing and subsequent heat firing.[3, 5]

Extensive research into the mechanical and optical properties as well as clinical performance of heat-pressed glass ceramics has been carried out over the past two decades.[6-18] In spite of this, only 4 studies that examined the mechanical, optical and microstructural properties of re-pressed ceramics were identified by the present author.

Albakry et al [3] **(2004)** evaluated the biaxial flexural strength (BFS) and identified the crystalline phases and the microstructural features of pressed and repressed materials of the glass ceramics (Empress I and Empress II). They concluded that the second pressing had no significant effect on the biaxial flexural strength of both glass-ceramics. However, higher strength variations among the repressed samples of the materials may indicate less reliability of these materials after second pressing.

Chung et al [19] **(2009)** studied the properties of four heat pressed glass-ceramic materials after repeated heat-pressing. Optimal pressable glass-ceramic OPC, 3G, Empress I and Empress II were evaluated. They stated that, repeated heat pressing treatment produced a statistically significant increase in the flexural strength of Empress II glass-ceramic material and Empress II secondary electron imaging (SEI) showed a densely packed, interlocking microstructure and an increase in size with preferred orientation of lithium disilicate crystals.

Gorman et al [5] **(2014)** investigated the effect of repeated pressing on the biaxial flexural strength, hardness and flexural toughness of lithium disilicate glass-ceramic (IPS e.max Press). They found no significant difference neither in BFS nor in flexural toughness but the hardness of the material decreased. Using x-ray diffraction analysis, lithium dislilicate was identified as the main crystal phase and no difference in crystalline composition was found with repeated pressing.

The phenomenon of light scattering largely affects the translucency of dental ceramics. If the majority of light passing through a ceramic is scattered, the material will appear opaque. However, if most of the light passing is transmitted through the ceramic it will appear translucent. [20] The amount of light that is absorbed, transmitted, and reflected mainly depends on the microstructure of the ceramic itself [21,22]. Differences in perceived color (ΔE) can be determined using the CIELAB coordinates. The CIELAB system has provided a quantitative representation of color and it has been extensively applied in dentistry to study esthetic materials, shade guides, and color reproductions. [23-25] The perceptibility and acceptability thresholds of the (ΔE) vary widely in literature mainly due to the diversity of observers, objectives, and methodologies among the studies [26, 27]. Clinically the tooth, restorations available, surrounding, and blending effect tend to expand the clinically acceptable range previously reported. [28, 29] The mean (ΔE) values as "clinically imperceptible" ($\Delta E < 3$), "clinically acceptable" (ΔE between 3 and 5) and "clinically unacceptable" ($\Delta E > 5$) seem to be consistent with the clinical practice considering a non-color expert, which usually is the patient's condition. [23, 30, 31]

Zaghloul et al [32] **(2013)** assessed the influence of repeated pressing on the color stability, translucency and surface roughness of three pressable glass-ceramics. IPS Empress Esthetic and IPS e.max Press and one zirconia veneering ceramic; IPS e.max ZirPress. They stated that, repeated pressing had a definite effect on the final color and translucency of the three tested pressable ceramic systems while, no significant influence on the surface roughness. The mean ΔE values caused by repeated pressing were below 3.7 units, which is rated as match in the oral environment. **They recommended that** further investigation should be carried out including microstructural analysis and x-ray diffraction (XRD) to evaluate the effect of second heat pressing on the color and translucency of the three tested glass-ceramics.

Few studies examined the mechanical [3, 5, 19] and optical properties [32] of repressed ceramics. From these studies, only **Albakry et al** [3] fabricated his samples by repressing the leftover buttons of lithium disilicate glass ceramics and discarded the leftover sprues. While the other studies; **Gorman et al** [5], **Chung et al** [19] and **Zaghloul et al** [32] repressed both leftover buttons and sprues to fabricate their samples.

Wang et al [33] **(2015)** in a study aimed to evaluate the influence of various heat-pressing procedures (different holding time and heat pressing temperature) on the microstructure and flexural strength of lithium disilicate glass ceramic. They stated that the presence of pores in the bulk or surface of dental ceramic have a detrimental influence on its flexural strength. Therefore porosity control (i.e. pore-free microstructure) should be a fundamental consideration during fabricating dental restoratives using glass ceramic, in order to reduce the frequency of fracture of dental ceramic restorations during function. This pore-free microstructure is mainly attributed to the densification effect of softened glass ceramic pressed at high temperature under vacuum. As a consequence of the loss of pressing effect, air bubble might be involved into the specimens. Porosity played a detrimental effect on the flexural strength by acting as a stress concentrator. This would be one of the factors contributing to lowering the flexural strength of heat-pressed glass ceramics.

Concerns whether repeated repressing affects the color reproduction, translucency and surface roughness of IPS e.max Press are still valid. Thus, the question is whether to reuse or not the left over material (sprues and button) is not yet answered.

The aim of the present study was to evaluate the effect of repressing and glazing on the color reproduction, translucency and surface roughness of lithium dislilicate glass-ceramics (IPS e.max Press). As well as to describe the microstructural features present in fresh-pressed, and repressed material using **Xray diffraction, EDAX and SEM**

MATERIALS AND METHODS

Twenty IPS e.max press discs (Ivoclar, Vivadent, Schaan, Liechtenstein) were prepared according to the ISO 6872 specifications for testing ceramic materials; discs of 15mm diameter, 1 mm thickness, in shade A3 were fabricated using heat pressed technique. A special custom made Teflon mold was constructed for this purpose. The discs were divided into two groups, Pressed group and Repressed group (n=10). For the Pressed group, discs were heat-pressed according to the manufacture's recommendation, then the leftover material was recovered where **Only** the button parts were adjusted by grinding to allow proper insertion into the refractory mold to construct specimens of re-pressed group by repeated heat-pressing. Pressing procedure used was the same for both pressed and repressed specimen groups. All discs were finished an glazed according to the manufacture's recommendation.

Color evaluation

Discs were placed over a neutral grey background and the CIELAB coordinates were measured for each specimen using an Easyshade spectrophotometer (Vita, Bad Säckingen, Germany). The ΔE was obtained after comparing the standard Vita coordinates of the A3 shade stored in the Easyshade. For each specimen three measurements were taken at the center and their average was recorded. After each specimen was measured the Easyshade was recalibrated. Mean ΔE values below 3.0 were considered "clinically imperceptible", ΔE values between 3.0 and 5.0 were considered "clinically acceptable" and ΔE values above 5.0 were considered "clinically unacceptable".

Translucency evaluation

A quantitative measurement of translucency was obtained by measuring the CIELAB coordinates of the discs after backing with a white and black background using the Vita Easyshade spectrophotometer. For each specimen three measurements were taken and their average was recorded. The contrast ratio (CR) for each specimen was calculated according to the following equation: $CR=Yb/Yw$ where $Y=[(L+16)/116]^3 \times 100$. In all calculations "0" was considered the most transparent and "1" was considered the most opaque.

Surface roughness evaluation

All discs were cleaned ultrasonically in 99% alcohol solution for 3 minutes then dried with air. The average surface roughness (Ra) for the specimens was measured using a 3D laser scanning microscope (Keyence VK-X100, Keyence GmbH, Neu-Isenbuerg, Germany). The wave length of the laser was 658 nm. Three separate areas were measured on each disc, the measured area was 500µm x 750µm and the distance between the separate scans was over 3µm. The mean Ra for each disc was later recorded before and after glazing.

Xray diffraction and EDAX

For each group, the discs were submitted to XRD to determine the crystalline phases. Samples were placed on the holder of the diffractometer (X pert pro, USA; PW 3040/60) and scanned using Cu Kα xray angle from 20-40 degrees, 2θ with a step size of 0.04 degrees and 5s-step interval. EDAX (energy dispersive x-ray analysis) was carried out to quantify elements by x-ray microanalysis (FEI Czech SEM - USA).

Microstructure by SEM

For each group, the discs were submitted to scanning electron microscopy (SEM). Samples were cleaned and etched with 9.8% Hydrofluoric acid for 90 seconds, cleaned in an ultrasonic cleaner, steamed, then dried and sputter coated with gold. SEM (Quanta 250 FEG) was carried out to examine the microstructure and assess grains at magnification of X 6000

Statistical Analysis

The data were collected, tabulated then analyzed using two-way analysis of variance (ANOVA), followed by Tuckey's HSD test at a significance level of $p< 0.05$.

RESULTS

Surface Roughness

Regarding surface roughness, for both groups, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups. No interaction between surface glazing and type of ceramic was found (Table-1).

TABLE (1) Mean Ra and standard deviation of test groups before and after glazing

Pressed				Repressed			
Glazed		Unglazed		Glazed		Unglazed	
Mean	SD	Mean	SD	Mean	SD	Mean	SD
0.27^{a}	0.05	1.14^{b}	0.16	0.21^{a}	0.06	1.33^{b}	0.20

Means with different superscript letters are statistically significant ($p<0.05$)

Colour Reproduction

Regarding Color reproduction, for both groups, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups (Table-2).

TABLE (2) Mean ΔE and standard deviation of test groups before and after glazing

Pressed				Repressed			
Glazed		Unglazed		Glazed		Unglazed	
Mean	SD	Mean	SD	Mean	SD	Mean	SD
3.40^{a}	0.20	5.72^{b}	0.22	3.48^{a}	0.13	5.76^{b}	0.23

Means with different superscript letters are statistically significant ($p<0.05$)

Translucency

Regarding Translucency, for both groups, significant difference was found between Glazed and Un-Glazed ceramics. No statistical significant difference was found between Pressed and Repressed groups (Table-3).

TABLE (3) Mean CR and standard deviation of test groups before and after glazing

Pressed				Repressed			
Glazed		Unglazed		Glazed		Unglazed	
Mean	SD	Mean	SD	Mean	SD	Mean	SD
0.59[a]	0.02	0.65[b]	0.03	0.60[a]	0.02	0.64[b]	0.02

Means with different superscript letters are statistically significant ($p<0.05$)

X-Ray Diffraction Analysis (XRD)

X-Ray Diffraction Analysis (XRD) investigates crystalline material structure, including atomic arrangement, crystallite size, and imperfections. The X-ray analysis (XRD) of both pressed and repressed samples detected diffraction peaks that correspond to crystalline phases present indicating that the material is predominantly crystalline structure; lithium disilicate was identified to be the main crystalline phase. Major peaks for lithium disilicate (Li2Si2O5) were observed at 2θ values of 24.7 degrees, 24.2 degrees, and 40 degrees. The dominant peak (highest peak) was at 24.7 degrees, which corresponds to the (040) crystallographic plane of this monoclinic phase, Corresponding to the standard peaks for lithium disilicate (Fig-1).

The XRD data showed that peaks after pressing and repeating pressing are similar, the crystalline phase assemblage did not change; however their radiation intensities (height) has, the dominant peak (highest peak) for the repressed sample is smaller compared to the pressed.

Microstructure by SEM

The SEM image observations (6000x); in pressed samples the length of lithium disilicate crystals averaged 3.05 μm in length while they averaged 473 nm in width, compared to repressed that averaged 6 μm in length while they averaged 500 nm in width. There is a noted increase in dimension after repressing (Fig-2).

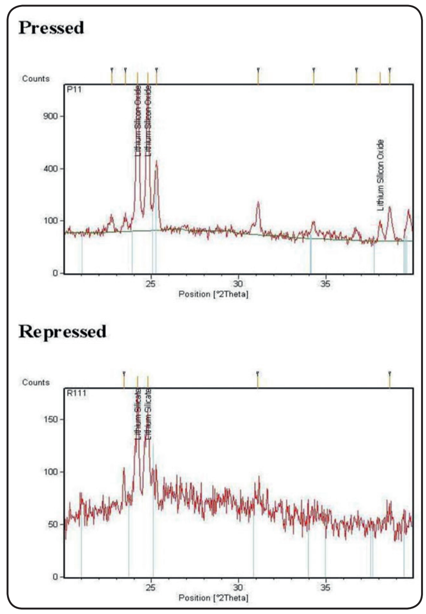

Fig. (1) X-ray diffraction (XRD) patterns of IPS e.max Press (Pressed) and (Repressed) showing peak positions in agreement with those of standard Lithium disilicate

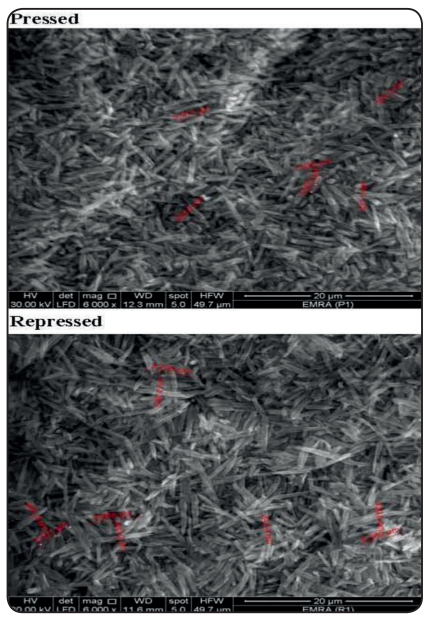

Fig. (2) SEM of IPS e.max Press (Pressed) and (Repressed) showing noted increase in dimension after repressing

EDAX

Energy Dispersive X-Ray Analysis (EDAX), is an x-ray technique used to identify the elemental composition of materials., it includes a lot of areas of applications. EDAX systems are attachments to Electron Microscopy instruments (Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) instruments where the imaging capability of the microscope identifies the specimen of interest. The data generated by EDAX analysis consist of spectra showing peaks corresponding to the elements making up the true composition of the sample being analyzed. It also allows elemental mapping of a sample and image analysis. It can be qualitative, semi-quantitative, and quantitative – it also provides spatial distribution of elements through mapping. The EDAX technique is non-destructive and specimens of interest can be examined in situ with little or no sample preparation. EDAX results showed no change in composition between pressed and repressed samples (Fig-3).

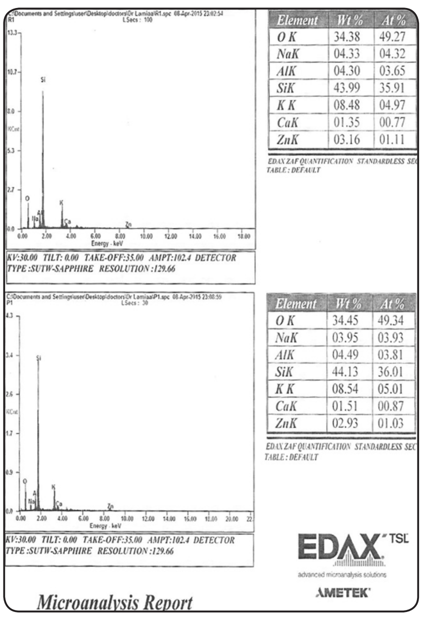

Fig. (3) Microanalysis by EDAX of IPS e.max Press (Pressed) and (Repressed)

DISCUSSION

Presence of pores in the bulk or surface of dental ceramic has a detrimental influence on the flexural strength as well as color reproduction and translucency of dental ceramics. [10, 33, 34] Therefore porosity control should be a fundamental consideration during fabricating dental restoratives using glass ceramic, in order to obtain optimal mechanical and esthetic outcomes.

In the present study, SEM scans (6000x) results for both groups (pressed and repressed) were found to be almost free of pores in the surface (Fig2). This pore-free microstructure was mainly attributed to using only the left over buttons and not using the left over sprues. This prevented trapping of air in-between the repressed material thus producing a repressed ceramic with nearly pore–free structure that is similar to that provided by the manufacturer and was used for the pressed group. These results **doesn't coincide** with **Chung et al** [19] who used both left over sprues and buttons to prepare the repressed samples. They found by SEM scans (5000x) multiple small pores located in the glassy matrix and at the lithium disilicate crystal grain boundaries, and had a higher etching rate than the lithium disilicate phase. They said that it is possible that the small pores observed by SEM in the microstructure of the lithium disilicate reinforced glass-ceramic were precipitates of Li_3PO_4 that may act as sites for the nucleation of stable lithium disilicate. They added that, there is a possibility of having an increase in porosity and crack because of multiple nucleation sites during crystallization.

The XRD and SEM data confirmed that the tested lithium disilicate-reinforced glass-ceramic materials contained lithium disilicate as a major crystalline phase, and that the amount of lithium disilicate did not increase as a result of repeated heat-pressing. Lithium disilicate elongated crystals were present in the glass matrix. They appeared to form an interlocking pattern in some sites; however, the lithium disilicate crystals in the repressed material were seen to be larger than those of the pressed samples. These results **coincide** with **Albakry et al** [3] **and Gorman et al** [5]**.** They stated that this behavior is called "Ostwald ripening" and is common for all precipitated materials. It takes place when the microstructure coarsens and liberates surface energy excess due to the solubility of small particles. As a consequence, larger grains are expected to grow at the expense of those small particles.

Color reproduction was not affected by repeated heat pressing but was significantly affected by glazing. For each group the mean ΔE values were above 5.0 which are considered “clinically unacceptable” but after glazing the mean ΔE values were between 3.0 and 5.0 which are considered “clinically acceptable”. This shows the importance of glazing to decrease surface roughness (Ra) that cause scattering of light and thus affecting the proper color production. [36] Repeated heat pressing on IPS e.max Press showed no effect on the color production ability. These results **doesn’t coincide** with **Zaghloul et al** [32]**.** The reported that second pressing resulted in more greenish IPS e-max Press specimens. They attributed this color change to the color instability of metal oxides during firing, which can affect the resulting color of ceramic. They used both leftover sprues and buttons to prepare the repressed samples which is not the case with our present study and hence the importance of pore–free structure for proper color production.

Translucency wasn’t affected by repeated heat pressing. The analysis in the present study revealed no changes were observed in glassy matrix and crystalline phase of IPS e.max Press heat pressed ceramics except slight increase in the size of lithium disilicate crystals and subsequently did not reveal negative effect on translucency parameter. These results **coincide** with **Albakry et al** [3]**, Chung et al** [19] **and Zaghloul et al** [32]**;** they stated that the microstructure of lithium disilicate glass-ceramic material might not have changed after repeated pressing.

Surface roughness (Ra) refers to the finer irregularities of the surface texture that usually result from the action of the production process or material condition and is measured in micrometers (μm) [35]. Surface roughness of IPS e.max Press was not affected by repeated heat pressing. These results coincide with **Zaghloul et al** [32]**.** They attributed there results to the fact that heat pressing is carried out under pressure from pneumatic press furnace. Besides, due to ingot delivery form, an improved density and homogeneity (porosity and bond) were achieved which prevent formation of defects within the ingot. Both resulted in defect-free and less porous specimens. Also, **Albakry et al** [3] who stated that no changes in the microstructure was found after repeated pressing, subsequently the second press has the same surface texture of the first one after finishing and glazing.

Regarding the effect of glazing; Color reproduction, Translucency and Surface roughness were affected significantly by glazing for both groups (Increasing color reproduction and translucency and decreasing surface roughness). This is due the fact that the purpose of glazing is to seal the open surface pores in the surface of fired porcelain, thus decreasing its surface roughness and decreasing light scattering. [25, 28-30, 36]

CONCLUSIONS

1- The microstructure of IPS e.max Press before and after repressing did not change. Indicating that lithium disilicate glass-ceramic materials have reached their maximum crystallinity prior to repressing.

2- It is important to produce a pore–free structure by only repressing the left over buttons and not the left over sprues.

3- Repressing has no significant effect on Color, translucency and surface roughness of IPS e.max Press.

4- Glazing is crucial to increase color reproduction, translucency and decrease surface roughness of IPS e.max Press.

Therefore according to the present investigation the IPS e.max Press leftover pressed buttons can be safely reused with no consequent negative effects related to Color, translucency and surface roughness. Further investigations, regarding surface treatment, cementation and bonding are recommended.

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