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PROPERTIES OF A NEW DENTAL PORCELAIN

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Abstract

A high strength dental porcelain OPTEC, HSP^{TM} has recently been introduced for use in the fabrication of crowns, three-unit anterior bridges with a single pontic, inlays, onlays, veneers, etc., without a metal sub-strate. This investigation reports the results of our research involving the characterization of the structure, properties and thermally induced transformations of this porcelain. It has been found that the material possesses superior strength and other properties relative to conventional porcelains. The improved properties appear to result from the presence of a very fine and relatively dense distribution of leucite crystals. Scanning electron microscopy indicates, however, that the new product needs additional optimization to improve uniformity of crystallite distribution. Scanning electron microscopy and X-ray diffraction studies also reveal well defined structural and crystallographic transformations during thermal treatment at selected temperature ranges. The primary transformation appears to be crystallization of metastable sanidine from the glass matrix. The crystallization of sanidine leads to a translucent to opaque transition. The transformations appear to follow the phase transitions expected from the phase diagrams.

<u>KEY WORDS</u>: Dental Ceramic, High Strength, Properties, Scanning electron microscopy, X-ray diffraction, Leucite, Sanidine.

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<u>Introduction</u>

A natural tooth reflects, disperses and transmits light through enamel and dentin, imparting an appearance of depth and vitality. To create the illusion of nature in a restoration, the selection of material to build up the restorations becomes the most demanding challenge. Dental porcelain with its characteristic translucency, is probably the material of choice to duplicate tooth in its subtleties of color, texture and form. (1,5,8,9,14) However, traditional porcelain used in dentistry lacks the necessary tensile strength for use by itself in stress bearing restorative applications. A metal substrate is used for porcelain build up so as to combine the benefits of metallic tensile and flexural properties with the translucency, color and aesthetics of porcelain. Even though such porcelain-fused to-metal (PFM) restorations have enjoyed a high reputation in fixed prosthodontics, a certain degree of com-promise is involved in the use of a metal substrate. The limitations stem from the following reasons:

- 1. Loss of translucency due to the use of metal substrate. This loss of translucency is particularly a shortcoming at the gingival margin.
- 2. Visible metal margin.
- Discoloration of porcelains due to the presence of silver in some alloys.
- 4. Decreased light transmission due to the metal substrate.
- 5. Potential corrosion of metal substrate and health hazard in some of the popular base metal alloys used as porcelain substrates. The long term effects of corrosion and release of cationic species into the biological system are not known. Some known allergenic and toxic elements like Ni and Be are used in popular PFM alloys. However, the presence of an intra-oral nickel appliance has been reported to reduce the likelihood of

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developing nickel allergy, (13) and the concentrations of beryllium used in dental alloys (less than 2%) have been demonstrated to be non-toxic in cell culture. (10)

A further drawback is the labor intensive nature of waxing, casting and finishing to obtain the alloy sub-strates, thus increasing the cost and time for the fabrication of restorations. The above limitations of PFM restorations have undoubtedly created searches for alternatives to optimize aesthetics, strength, biocompatibility and cost effectiveness. The initial outcome of these searches is the introduction of a first generation of high strength ceramic systems such as Dicor (Dentsply Inc., York, PA)² Cerapearl (Kyo Ceram Inc., San Diego, CA),³ aluminous core materials (Vita Zahnfabrik, Bad Sackingen, W.G.), OPTEC, HSP[™] (Jeneric Pentron Inc., Wallingford, CT), etc. While each of these systems has its merits, the focus of this investigation is an in vitro evaluation of the leucite-strengthened porcelain called OPTEC, HSP[™]. Since the system uses conventional fabrication procedures in the dental laboratories, there is a distinct advantage of accept-ance by the dental laboratories, as no added capital outlay is involved.

In this study, OPTEC, HSP^{IM} was evaluated for mechanical properties, microstructures and thermally induced transformations.

Materials and Methods

Porcelain frit supplied with OPTEC, HSPTM kit from Jeneric Pentron Inc. was moistened, packed in appropriate molds and compacted with a Carver Press under 1500 kg load. The compacted samples were fused in an automatic porcelain furnace (Ney Sunfire) by raising the temperature at 55°C/min from 650°C to 1035°C in 720mm vacuum. The samples were held for 2 minutes at 1035°C and bench cooled. They were tested for the different properties using the specimens and instruments indicated in table 1.

Porcelain was characterized by scanning electron microscopy and X-ray diffraction. Disk samples were also prepared as described previously for scanning electron microscopy, X-ray diffraction studies and thermal processing studies. Scanning electron microscopy was carried out in a Hitachi SEM (Scanning Electron Microscope) model S-2500 interfaced to a Kevex EDS (Energy Dispersive Spectroscopy) and advanced imaging system. The surfaces were slightly etched with a 1% HF acid solution for 20 sec and sputtercoated with gold prior to SEM evaluation. The XRD (X-ray diffraction) studies were carried out in a Philips or Rigaku X-ray diffractometer (Copper, K α , 45kV, 35mA). Thermal processing of the disk samples included isothermal treatment by holding the fused porcelain disk for 1 hr. at 650°C, 705°C, 760°C, 815°C, 870°C, 925°C and 1/2 hr. at 980°C. The thermally processed samples were visually examined and characterized by scanning electron microscopy and X-ray diffraction.

Experimental Results and Discussion

<u>Properties of OPTEC, HSP</u>™:

Table 2 lists the mechanical and physical properties measured for OPTEC, HSP^{IM}.

Generally, the mechanical proper-ties (compressive strength and modulus of rupture) were found to be superior to the reported properties for conventional porcelains. For example, the reported modulus of rupture of conventional porcelain is significantly lower at 55-75 MPa.⁽¹¹⁾ Impact strength values were, however, nearly equal at around 5 kg.cm. The high thermal expansion value of 0.899% at 500°C observed for OPTEC, $\mathtt{HSP}^{\mathtt{IM}}$ sample is attributed to arise from the presence of a large volume fraction of leuctie in the structure. The improvement in the mechanical properties can be explained by the strengthening effect associated with a fine dispersion of a second phase. It is believed that the large thermal expansion mismatch be-tween leucite $(20-25\times10^{-6}/^{\circ}C)^{4,7}$ and the glass matrix $(8\times10^{-6}/^{\circ}C)$ result in stress fields within and around leucite particles during cooling. The stress field created by a higher expanding particle (leucite) in a lower expanding matrix (glass) is compressive in the tangential direction of the particlematrix interface and tensile in the radial direction. Such stress field is believed to strengthen the glass ceramic system, resulting in the observed improvement in selected properties.

Frit Characterization

Figure 1 is a scanning electron micrograph of OPTEC, HSP^{IM} porcelain frit supplied by the manufacturer in the original kit. Figure 2 is a segment of the XRD profile of the above frit in the O° to 37°, 20 range. XRD profile of a conventional feldspathic porcelain frit (Pencraft), obtained under identical scan range and conditions is given in figure 3. Search and match software identified the XRD spectral peaks (of both the OPTEC and PENCRAFT frits) to be that of leucite. In addition, the presence of the amorphous glass phase is also apparent from the difuse background. Table 3 lists the ratios of



Fig. 1: Microstructure of OPTEC, HSP Frit

selected leucite XRD spectral peaks in the conventional frit to that in OPTEC frit. It is apparent from the values of intensity ratio I(conv.)/I(optec) in table 3 that OPTEC, HSP^{IM} porcelain frit contains approximately 60% more leucite than the conventional frit.

<u>Microstructures and XRD Study of Fused</u> <u>Porcelain</u>

Figures 4 and 5 show the scanning electron micrographs of selected regions of a fused porcelain disk. A second phase dispersion in a matrix phase is clearly visible in the microstructures. The second phase particle sizes range from submicron size to 20µm or more. However, most of the second phase particles appear to be in the 1 - 10µm range and a considerable fraction in the 1 - 5µm range. The particle dispersion also ranges from dense dispersion in some micro-regions to lighter dispersion with larger interparticle separation in other micro-regions (see Figures 6a to 6d). Analysis by Kevex feature analysis software showed that the particle volume fraction ranged from 20 to 55% in different imaging areas. Thus, the second phase distribution appears to be nonuniform and clearly needs improvement. In addition, fracture lines were observed in some part of the microstructures (Figure 7). Such non-uniform dispersion and appearance of fracture lines probably result from the specific manufacturing process used. Porcelain manufacturing process used. Porcelain



Fig. 2: XRD Profile of OPTEC, HSP Frit



Fig. 3: XRD Profile of PENCRAFT Frit

frit is prepared by mixing potash feldspar with KNO3, K20.SiO2, Li2CO3, and cerium oxide in CaCO3, MgCO3 specified ratios and raising the temperature of the mix to a specific time The interval at that temperature. soaked mass is then cooled to an intermediate temperature, quenched in water, dried, pulverized and ball milled. Although the product contains about 60% more leucite than the conventional feldspathic dental porcelain frit, the manufacturing process is not presently optimized to disperse the second phase uniformly, densely and in optimal particle size distributions.

Figure 8 shows an XRD profile and clearly identifies the second phase to be leucite. The four peaks at 25.9, 27.2, 30.6 and 31.5° 20 values correspond to the expected high intensity peaks for leucite in the scanned range. A spot spectrum by CARD (Composition Analysis by Robinson Detector) and identification by effective atomic number confirmed the crystalline phase to be leucite (see Figure 9).

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s.	# Property	Specimen Size and Geometry	Sample size(#)	Test Setup
	Thermal Expansion	Disks(6mm dia x 3mm thick)	3	Dupont Thermomechanical Analyzer
	lmpact Strength	6mm x 6mm x 50mm bars (V notch)	10	Izod Test Setup (Testing M/C Inc.)
	3 Modulus of Rupture	4mm x 6mm x 50mm bars (All sides ground flat on emery paper, three sides given a wash of glaze)	10	Three point bending in an Instron Universal Testing Machine
	Compressive Strength	4mm dia x 8mm thick Cylinders	10	Instron Universal Testing Machine
	Vicker's Hardness Number (VHN)	25mm dia x 6mm thick disks	10	Clark Microhardness Tester

TABLE 1: Specimen and Experimental Setup Details

TABLE 2: Properties of OPTEC, HSP

S.No.	Property	Mean(SD)	Units
<u> </u>	Thermal Expansion(30-500°C)	0.899(0.015)	<u>o</u>
2	Modulus of Rupture	145.9(16.56)	MPa
3	Compressive Strength	965.9(54.52)	MPa
4	Impact Strength	4.95(0.3)	Kg.Cm.
5	VHN(Vicker's Hardness Number)	402(14)	_

TABLE 3: Ratios of Intensities of Selected Leucite XRD Peaks of Conventional Frit vs. Optec Frit

S.No.	20 (deg.)	(Å)	l _{conv} (counts)	loptec (counts)	I _{conv} . I _{optec}	Remarks (calculated increase in leucite)
1	16.45	5.389	581	942	0.62	618
2	25.93	3.436	1,444	2,421	0.60	67%
3 :	27.21	3.277	1,875	3,192	0.59	69%
4	31.43	2.846	630	1,109	0.57	75%
5	31.79	2.815	380	605	0.63	59 %
6	37.95	2.371	408	666	0.61	648

Thermal Processing Effects

Visual examination of the thermally processed disks revealed that thermal processing of the samples at 650° C and 980° C showed no changes in translucency. However, thermal treatment at 705° C, 760° C, 815° C, 870° C and 925° C resulted in a loss of translucency and the samples became opaque. SEM analysis revelaed that while the leucite dispersed structure was retained after thermal processing at temperatures of 650° C and 980° C, an additional crystalline phase was detected at 705° C through 925° C. Figures 10-16 show representative microstructures observed across the translucent-opaque-translucent transition temperatures. The new crystalline phase detected in the opaque samples revealed an interpenetrating rod-like morphological appearance. Xray diffraction confirmed that leucite was the only detectable crystalline phase in samples processed at 650°C and 980°C. However, samples processed at 705°C, 760°C, 815°C, 870°C and 925°C all produced XRD peaks typical of a second crystalline phase in addition to that of leucite. Figures 17 and 18 show XRD profiles of samples thermally processed at 980°C and 760°C, respectively. The XRD profiles at 980°C indicate peaks at 25.9, 27.2 and 30.4°, 20 values and can readily be identified to correspond to the d values of 3.436, 3.277 and 2.92Å for leucite from JCPDS file #25-47. On the other hand, the thermally processed sample at 760°C shows three additional

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Fig. 4: Microstructure of fused OPTEC, HSP



- Fig. 6a: Illustration of a dense distribution of leucite crystals in a selected region
- Fig. 6b: Marked region in 6(a) magnified five times

peaks not observed in the sample processed at 980° C. These peaks occur at 20 values of 26.9° , 27.6° and 29.8° , respectively and correspond to d values of 3.27, 3.26 and 2.98Å, the three expected high intensity lines for



Fig. 5: Higher magnification microstructure of a selected region



- Fig. 6c: Illustration of sparse, coarse distribution of leucite in some region
- Fig. 6d: Marked region in 6(c) magnified five times

sanidine. Thus, it is reasonable to conclude that the translucent-opaquetranslucent transition is associated with the crystallization of metastable sanidine phase and the temperature range across which this phase is found to be



Fig. 7a: Illustration of fracture lines in OPTEC, HSP microstructure
Fig. 7b; Marked fracture region in 7(a) magnified five times

The ratios of the sanidine peak stable. (27.6° and 29.8°, 20) intensities to leucite peak (25.9°, 27.2° and 30.4°, 20) intensities vs. firing temperatures are shown in Figure 19. The formation and disappearance of the sanidine phase across the translucent-opaque transition range of temperatures of 650°C-980°C is clearly evident. In addition, the maximum sanidine appears to form in the 760-870⁰C temperature range with the highest transformation probably occuring at 760⁰C. Moreover, the relative absence of change in the intensity levels of leucite peaks across the translucencyto-opaque transition range of temperatures indicated that sanidine formation does not occur at the expense of leucite phase, but rather through crystal-lization of sanidine from the glass matrix. This is, however, to be expected because the glass and sanidine compositions are stoichiometrically nearly identical to (Na,K) 20. Al 203. 6Si02 in this system. The formation and disappearance of sanidine phase across a temperature range observed in this study can be readily explained by an examination of the Albite $(Na_20^{\circ}Al_2O_3^{\circ}6SiO_2)$ - Orthoclase $(K_20^{\circ}Al_2O_3^{\circ}6SiO_2)$ phase diagram⁽¹²⁾ shown in Figure 20. It can be seen that sanidine is a metastable phase in the temperature range of about 500° -925°C. This range nearly coincides with the temperatures where sanidine forma-tion is observed in this study, except



Fig. 8: XRD Profile of fused OPTEC, HSP



Fig. 9: Spot spectrum from CARD. Note the superposition of the spectral peak with the cursor position at 12.2 corresponding to the atomic number factor of leucite

at the lower temperature range where additives used in the formulation have probably shifted the range of metastability to higher values of temperatures of $>650^{\circ}C$.

The identification of a translucent to opaque optical transformation in the thermal processing also needs some examination. In spite of the higher volume fraction of leucite phase in OP-TEC, HSP^{IM} (as compared to conventional dental porcelains), the product appears to retain its translucency. This is readily attributed to the closeness of the refractive index of leucite $(n=1.509)^{(6)}$ to that of glass matrix $(n=1.505\pm0.004)$. However, formation of sanidine in the $705^{\circ}-925^{\circ}C$ range results in loss of translucency.





Fig. 12: Microstructure after 1 hr. at 760°C

<u>Conclusions</u>

The mechanical properties of OPTEC, HSP^{IM} appear to be superior to those of conventional dental porcelain. The in-



Fig. 11: Microstructure after 1 hr. at 705°C



Fig. 13: Microstructure after 1 hr. at 815°C

crease in strength is probably the result of a dispersed second phase of leucite crystals over a wide spectrum of particle sizes and distribution. OPTEC, HSP^{IM} system undergoes optical, crystallographic and morphological changes in its structure in the 650-980°C temperature range. The primary transformation occurs through the change of glass matrix to sanidine phase.



Fig. 14: Microstructure after 1 hr. at 870°C



Fig. 16: Microstructure after 1/2 hr. at 980°C

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Fig. 15a: Microstructure after 1 hr. at 925°C Fig. 15b: Marked region in 15a magnified

five times



Fig. 17: XRD Profile after 1/2 hr. at 980°C

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XRD_Profile after 1/2 hr. at Fig. 18: 760⁰C



Fig. 20: Albite - Orthoclase Phase Diagram

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Ratio of Sanidine to Leucite Lines



Variation of the ratios of Fig. 19: sanidine to leucite intensitites of selected peaks as a function of temperature

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Discussion with Reviewers

<u>Reviewer 1</u>: The authors use ratios of peak height intensities to derive information about relative concentrations of leucite in OPTEC versus a conventional While peak heights are porcelain. proportional to concentrations, peak broadening caused by particle size effects and other factors will cause the peak height to be smaller even when the concentration is the same. The authors should use the integrated intensities with background subtracted out to compare the relative concentrations of leucite in the two porcelains.

Authors: The total integrated area of five leucite peaks in the $21 - 28^{\circ} 29$ range, after correction for background, was determined to be 54,051 counts for OPTEC. The corresponding integrated area for PENCRAFT was 29,081. The ratio of I_{PENCR} to I_{OPTEC} was 0.54 based on these measurements. While this indicates 80% more leucite in OPTEC than in PENCRAFT, our data in the text indicate a conservative estimate to a reasonable degree of accuracy sought by us. More accurate determination can be made by statistical and quantitative techniques, but our objective was more limited in this investigation.

<u>Reviewer 2</u>: The shrinkage of OPTEC porcelain compromises the fit of restorations at marginal areas compared with a metal margin. Firing shrinkage of this product requires one or more extra firings to achieve equivalent marginal fit. Since this deficiency requires extra laboratory time, the labor intensive process of metal preparation of PFM restorations may not increase the cost appreciably compared with the OPTEC cost. This economy should be substantiated. Another benefit of PFM crowns is their success rates, i.e., sucess rates of 97% for crowns and bridges over a 7.5 year period (Coornaert). OPTEC HSP has no such track record of longevity.

<u>Authors</u>: There is no question in our minds that PFM restorations are the standards against which all-ceramic restorations have to be clinically judged, but the jury on this judgment is still out until clinical performance evaluations of the product are completed. Our studies, collaborated by dental laboratory evaluations, indicate a minimum of 15 - 20% savings in the fabrication time for equivalent marginal fit. The shrinkage of OPTEC is only slightly higher than that of porcelain for PFM. If a thick layer of opaque porcelain is fired onto a metal substrate, even the conventional porcelain will have the tendency to delaminate or lift up at the margins. Similarly, if a thick layer of OPTEC porcelain is applied on to a refractory die substrate and not fired properly, a tendency for lift up is to be expected. However, this problem is circumvented by the application of a very thin band at the margin in the initial bake. The marginal integrity of OPTEC is also dependent on the proper refractory powder to liquid ratio. Similarly, the marginal integrity of a metal substrate in PFM is dependent on the proper ratio of investment to liquid ratio. In both systems, once a proper

ratio is determined, identical marginal integrity can be obtained.

<u>Reviewer 2</u>: The modulus of rupture (MOR) of the OPTEC HSP specimens cannot be determined with a "wash of glaze" applied. Why are the MOR values not clearly associated in the Discussion section for altered (glazed) OPTEC, HSP specimens?

Authors: The three point bending test methodology used for the MOR determination has not been standardized and universally accepted at the present time. A discussion of the various processing procedures for the test have been summarized in a report (Feb 4, 1988) by the R & D department, Ivoclar-Vivadent. Apparently, different inves-tigators have used different methods for the sample preparation. We recognize the potential variability due to the method of sample preparation. According to the ISO procedures recommended for jacket crown ceramic, grooves produced in the grinding process are smoothed with a glaze firing (DIN- Vorlage 0013925, January 1987: Dental-Keramische Massen; International Standards Organization, standard 6872, 1984: Dental Ceramic). The wash of glaze in our test was done at a temperature of 980° C, which is 50° C lower than the firing temperature of OPTEC. We have carried out additional tests according to the ISO standard and obtained a mean MOR value of 133.44 (22.03) MPa using a sample size of n=7.

<u>Reviewer 2</u>: The implication of strengthening due to fine dispersion of a second phase has not been proven in this study. Why should a second phase necessarily strengthen the ceramic? What is the influence of other factors such as stresses or microcracks around the particles due to large thermal contraction differences?

<u>Authors</u>: We have demonstrated that there is a high volume fraction and small particle distribution of leucite crystals in the OPTEC structure. Both SEM and XRD studies support this conclusion. We show that there is at least a 60% higher leucite content in OPTEC than in the conventional porcelain control. Davidge and Green (Davidge RW, Green TJ (1968) The strength of two phase ceramic materials. J Mater Sci, 3, 629-634.) have discussed the strength of two phase ceramic materials and indicated that the important factors influencing the strength are:

- 1. The expansion coefficient of the two phases
- 2. The elastic properties of the two phases

3. The volume fraction of the crystalline phase metric composition.

4. The particle size of the crystalline phase

While the relative contributions of these factors is not yet known, it is only reasonable to conclude at this stage that these factors are important in the strengthening of OPTEC porcelain. Clearly, additional systematic work is needed to establish the interactive effects of these factors in the strengthening of this system.

<u>Reviewer 2</u>: Has a quantitative analysis been performed to calculate the particle size, volume fraction and particle size distribution?

<u>Authors</u>: We plan to carry out a systematic investigation on leucite volume fraction, particle size and distribution and their influence on properties in future.

<u>Reviewer 2</u>: Has translucency been measured? Visual examination is not acceptable since the observers were not calibrated.

<u>Authors</u>: It is important to realize that this study was not focused on the topic of translucency. Measurement and observer calibration are important only when a range of translucency values are involved. In our opinion, differentation of opaque and translucent specimens do not require detailed measurements. Qualitative differentiation of opaque samples from translucent samples was not complex.

<u>H.J. Mueller</u>: Is it likely to manufacture porcelains with even higher concentrations of leucite than in OPTEC, HSPTM?

<u>Authors</u>: Yes, but there will be an increase in the fusion range and it is not known what effect such increase in volume fraction will have on the translucency.

<u>H.J. Mueller</u>: If potash feldspar is mixed with other ingredients, where does the sodium come from? <u>Authors</u>: Potash feldspar usually contains some albite.

<u>H.J. Mueller</u>: Are there chemical or structural differences between leucite and sanidine?

<u>Authors</u>: Our studies indicate an interpentrating rod-like morphology for sanidine. From the phase diagram in figure 20, it is also clear that the chemical composition of sanidine may vary. The crystal structure observed for sanidine is monoclinic. Leucite can crystallize in the tetragonal or cubic polymorphs and has a definite stoichio-