

Charges extracted from dental ceramics: An indication of piezoelectric activity?

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A method was established for demonstrating charge generation on the surfaces of dental materials by means of mechanical loading with different forces. With this experimental set-up charges could be extracted both from the surfaces that were mechanically loaded and from the surfaces perpendicular to the applied force. When specimens of certain ceramics were subjected to mechanical loading and the charges extracted were related to the applied force, charges of up to $3 \cdot 10^{-3}$ pC/N were recorded from the surfaces perpendicular to the applied force and charges of up to $8 \cdot 10^{-3}$ pC/N from the surfaces that were mechanically loaded. The results from this study indicate support for the hypothesis that the extracted charges were created by direct piezoelectricity. However, further studies are necessary for decisive proof that true piezoelectricity occurs in these materials. □ *Ceramic inlays; dental materials; dental porcelain*

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In a previous study direct piezoelectricity was proposed (1) as a conceivable cause of the sharp, transient, clearly localized pain on loading reported in connection with posterior ceramic or resin inlays (2, 3). In that study (1) it was shown that current could be extracted when specimens of certain dental ceramics were subjected to dynamic forces. The purpose of the study (1) was to examine whether the direct piezoelectric effect ever occurs in some materials intended for dental use. The test arrangement was designed to demonstrate *currents* extracted from electrodes on surfaces perpendicular to the direction of an applied *dynamic* force (the transverse mode). Since it is the longitudinal mode—that is, when the charge appears on the *loaded* surfaces—that should be most important clinically, it was of particular interest to study the possibility of demonstrating charge generation on those surfaces. Furthermore, since the Greek word *piezein* means 'to press', it was interesting to investigate whether charges could also be created by means of a slowly varying loading on dental ceramics.

The aim of the present work therefore was to establish a method whereby the generation of charges created on the loaded surfaces and on the surfaces perpendicular to the direction of an applied force could be demonstrated.

Materials and methods

Two different dental ceramics, Dicor-MGC Glass-Ceramic (Dicor, De Trey Dentsply Int., Wiesbaden, Germany) and Cerec Vitablocs Mark II (Vita Zahnfabrik, H. Rauter GmbH & Co KG, Bad Säckingen, Germany), were studied. The ceramics were delivered as prefabricated ceramic blocks intended for machining

by means of the CAD/CAM technique. Twelve specimens of each brand were studied. Both the Cerec Vitablocs specimens and the Dicor-MGC specimens came from two different batches.

Longitudinal mode

To study the longitudinal mode, the ceramic block was separated into two equal parts by a cut parallel to its bottom surface. The upper part was then turned 180° and fixed on top of the lower part with copper foil tape coated with conductive adhesive on both sides (Scotch Electrical Tape 1182, 3M Electrical Specialties Division, Austin, Tex., USA). In this manner the top surface of the upper part of the original prefabricated ceramic block was placed against the surface of the top of the lower part of the divided ceramic block (Fig. 1). The specimen measured (14 × 12 × 10) mm. The copper foil tape that separated the specimen halves served as signal electrode for extracting charges from the specimen.

The specimen was placed in an aluminum can, which served as a Faraday cage. A steel plate, 4 mm thick, was screwed on the bottom of this aluminum can. A 1-mm-thick piece of tinfoil was fixed to the steel plate with a copper tape with conductive adhesive on both sides (Scotch Electrical Tape 1182). The specimen was then fixed on the top of the tinfoil by means of more copper foil tape with conductive adhesive on both sides (Scotch Electrical Tape 1182). This copper foil tape served as grounding electrode for the lower half of the specimen.

Subsequently, a spiral copper wire (diameter ≈ 15 μm) was soldered to the copper foil tape in the middle of the specimen—that is, the signal electrode. Another spiral copper wire (diam-

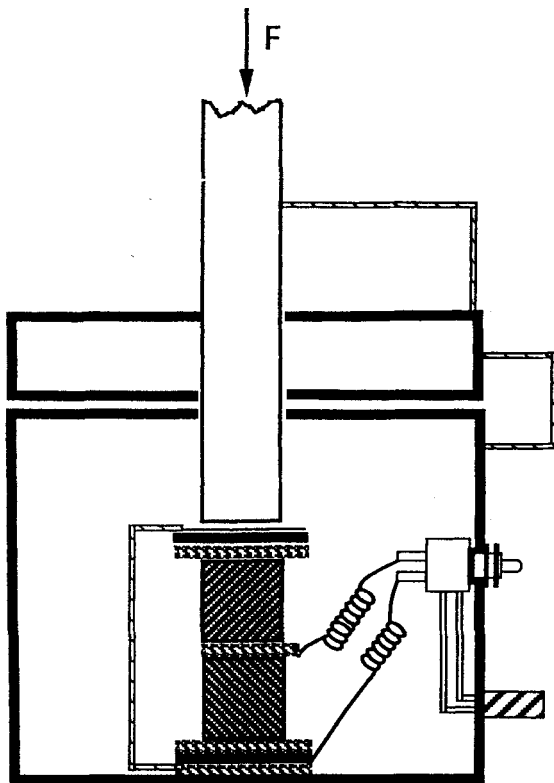


Fig. 1. The Faraday cage containing a test specimen for the longitudinal mode testing. F = force; (▨) = specimen; (▧) = shielded cable; (▩) = tinfoil; (▪) = grounding cable; (▬) = copper foil tape with conductive adhesive on both sides; (▮) = copper foil tape with conductive adhesive on one side; (⌀) = spiral copper wire with a diameter of 15 μm ; (⊗) = polarity switch.

eter $\approx 15 \mu\text{m}$) was soldered to the copper foil on the bottom of the aluminum can, the grounding electrode (Fig. 1). The opposite ends of these electrode wires were then connected, via a polarity switch, with the input of a charge amplifier designed and constructed at the Laboratory of Electronics, Faculty of Medicine, Umeå University, Umeå, Sweden, by one of the authors. This charge amplifier detected charges generated by the specimen and did not itself inject charges to the specimen and had outputs for both charge and current and a low-input impedance ($\approx 1 \text{ k}\Omega$). The charge amplifier was supplied with stabilized mains power, and the output was connected to an oscilloscope (Advanced Instruments, Advance Electronics Ltd, Essex, England). All connections between the oscilloscope, the amplifier, and the Faraday cage were conducted through shielded cables.

On the top surface of the specimen a square-shaped piece of copper foil tape with conductive adhesive on both sides (Scotch Electrical Tape 1182), measuring

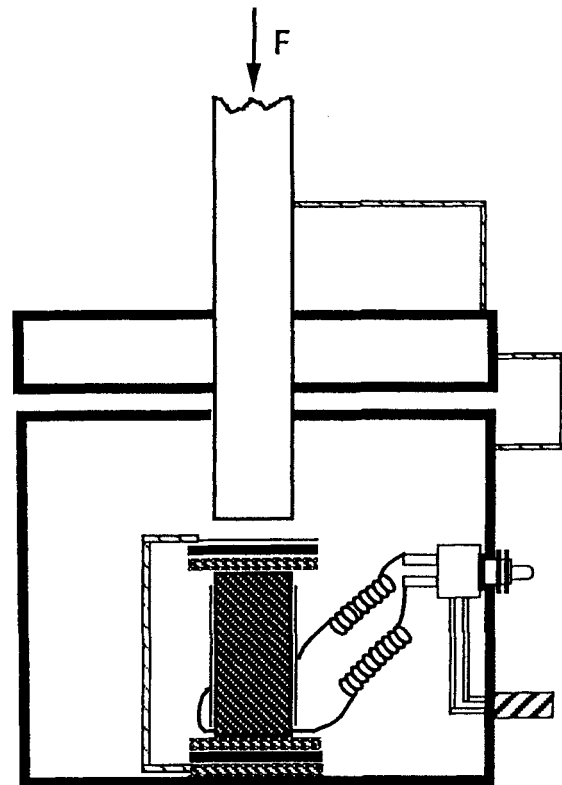


Fig. 2. The Faraday cage containing a test specimen for transverse mode testing. See Fig. 1 for key to symbols.

(14 \times 16) mm, was fixed. On top of this copper foil tape a piece of tinfoil, 1 mm thick, was placed, and on the top of that was fixed a copper foil tape coated with conductive adhesive on one side (Scotch Electrical Tape 1181). These copper foil tapes and tinfoil were all electrically connected to ground through a cable soldered to the copper foil tape on the bottom of the aluminum can. Thereafter a steel rod was inserted through a hole in the lid of the aluminum can. The end of the steel rod placed inside the aluminum can was square, measuring (12 \times 10) mm, and the opposite end was fixed to an Alvetron TCT 5/10 testing machine (Lorenzen and Wettre, Spånga, Sweden). The aluminum can, the lid of the aluminum can, and the steel rod were all electrically grounded. After this the square end of the steel rod was maneuvered into mechanical contact with the top of the specimen with the electrically grounded copper foil tapes and the piece of tinfoil placed between the ceramic surface and the steel rod (Fig. 1).

To enable, as far as possible, an even distribution of the applied force on the loaded surfaces, the force was first cycled 50 times through the range of 5500 N and 100 N at a frequency of 0.6 Hz by means of the Alvetron testing machine. Thereafter the specimen was

Table 1. Mean values, standard deviations, and ranges of charges extracted (pC) in relation to applied force (N); $n = 6$ for each type of specimen

Type	Extracted charge/force (pC/N)	Range of extracted charge/force (pC/N)
Cerec Vitablocs, transverse mode	$5.0 \cdot 10^{-4} \pm 7.5 \cdot 10^{-4}$	$(1.1 \cdot 10^{-4} - 2.7 \cdot 10^{-3})$
Dicor MGC, transverse mode	$1.1 \cdot 10^{-4} \pm 0.8 \cdot 10^{-4}$	$(\approx 0 - 2.9 \cdot 10^{-4})$
Cerec Vitablocs, longitudinal mode	$2.0 \cdot 10^{-3} \pm 2.6 \cdot 10^{-3}$	$(1.8 \cdot 10^{-4} - 7.9 \cdot 10^{-3})$
Dicor MGC, longitudinal mode	$1.4 \cdot 10^{-4} \pm 1.0 \cdot 10^{-4}$	$(\approx 0 - 4.0 \cdot 10^{-4})$

loaded by means of the testing machine with a force cycled, at a frequency of 0.6 Hz, through the ranges 1100 N–100 N, 2100 N–100 N, 3100 N–100 N, 4100 N–100 N, or 5100 N–100 N, respectively, and the extracted charge was recorded from the oscilloscope tracing. These loadings were repeated three times for each range of force, and the mean values of the charge extracted were calculated. The calculated charge was the charge difference observed within each range of force—that is, the charge generated when the specimen was loaded from 100 N to the maximum level of the applied force within the selected range.

Transverse mode

To study the generation of charges on the surfaces perpendicular to the direction of an applied force, the transverse mode, copper foil tape with conductive adhesive on one side (Scotch Electrical Tape 1181) was fixed on two opposite surfaces of the specimen. The specimen measured (15 × 12 × 10) mm. Spiral copper wires (diameter ≈ 15 μm)—that is, the electrode wires—were soldered to the copper foil tapes, one wire on either side of the surfaces with the copper foil tape (Fig. 2). These electrode wires were connected with the charge amplifier input by means of the polarity switch. The rest of the experiments were carried out in the

same manner as in the test for the longitudinal mode. In addition, the effect of changing which of the two electrodes was connected to the signal electrode or the signal-ground electrode of the charge amplifier was studied by shifting these connections by means of the polarity switch.

All the values calculated from the oscilloscope tracing for the longitudinal mode were divided by 2, since the charges were extracted from two surfaces and, with regard to the dimension of the specimen, the values calculated for the transverse mode were divided by 1.5, the form factor (4).

Results

The mean values, the standard deviations, and the ranges of extracted charge in relation to applied force are shown in Table 1. In Figs. 3–6 extracted charge is plotted versus applied force. As can be seen in Figs. 3–6, the relationship between applied force and extracted charge is fairly linear.

For one of the six Cerec Vitablocs specimens the magnitude of the charge extracted for the transverse mode differed considerably from that for the other five Cerec Vitablocs specimens studied. The mean value for

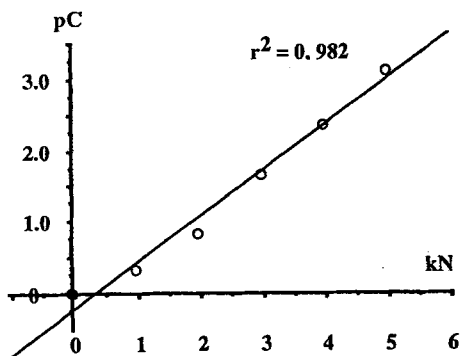


Fig. 3. Mean values of extracted charge (pC) versus applied force (kN) in the transverse mode testing of the Cerec Vitablocs specimens; $n = 6$.

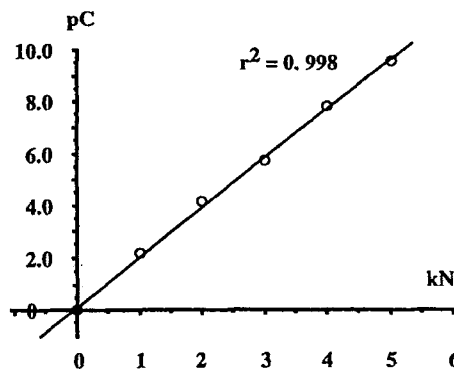


Fig. 4. Mean values of extracted charge (pC) versus applied force (kN) in the longitudinal mode testing of the Cerec Vitablocs specimens; $n = 6$.

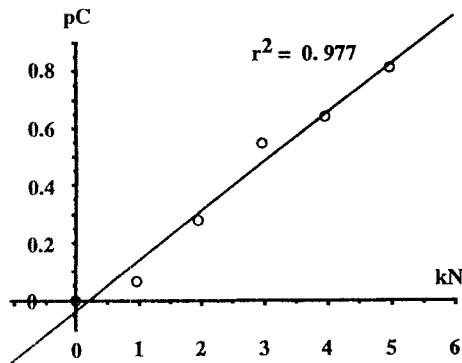


Fig. 5. Mean values of extracted charge (pC) versus applied force (kN) in the longitudinal mode testing of the Dicor MGC specimens; $n = 6$.

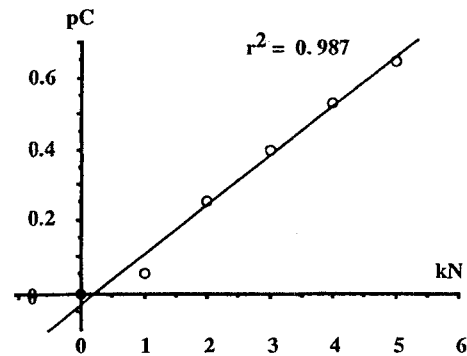


Fig. 6. Mean values of extracted charge (pC) versus applied force (kN) in the transverse mode testing of the Dicor MGC specimens; $n = 6$.

the extracted charge related to applied force, calculated from the loadings at each range of force, for this divergent specimen was $2 \cdot 10^{-3} \pm 0.7 \cdot 10^{-3}$ pC/N (range, $1.2 \cdot 10^{-3}$ pC/N– $2.7 \cdot 10^{-3}$ pC/N), compared with $2 \cdot 10^{-4} \pm 0.5 \cdot 10^{-4}$ pC/N (range, $1.1 \cdot 10^{-4}$ pC/N– $2.9 \cdot 10^{-4}$ pC/N) for the other five specimens.

For the longitudinal mode the magnitude of the extracted charge for three of the Cerec Vitablocs specimens was considerably higher than for the other three specimens in this group. The mean value for the extracted charge in relation to applied force of the three specimens that showed the higher magnitudes was $3.7 \cdot 10^{-3}$ pC/N \pm $2.9 \cdot 10^{-3}$ (range, $0.9 \cdot 10^{-3}$ pC/N– $7.9 \cdot 10^{-3}$ pC/N), compared with $3.3 \cdot 10^{-4}$ pC/N \pm $0.8 \cdot 10^{-4}$ (range, $1.8 \cdot 10^{-4}$ pC/N– $5.2 \cdot 10^{-4}$ pC/N) for the three specimens that showed the lower magnitudes.

When the charge was extracted from the specimen surfaces during the transverse mode testing, the oscilloscope trace changed direction when the connections between the electrodes and the charge amplifier were shifted by means of the polarity switch. Recorded magnitudes remained unchanged.

Discussion

Direct piezoelectricity means that an electric charge is generated on the surfaces of certain anisotropic crystals when a mechanical force is applied (5–7). Since fired ceramic products may inherit a textural anisotropy (8) and it has been observed that heating certain ceramics to 950°C can result in a highly anisotropic growth of the crystals (9), it was of interest to establish methods that would make it possible to study whether direct piezoelectricity occurs in certain dental ceramics. This was interesting because one of the postoperative symptoms reported in connection with posterior ceramic inlays is a sharp, transient clearly localized

pain on loading of the inlays (2, 3), and the possibility cannot be excluded that electricity may be one of the causes of this symptom.

The results of the present study showed that charges could be extracted from the surfaces of the dental ceramics studied when the specimens were subjected to loading. Charges were extracted both when the electrodes were placed on the surfaces that were loaded, the longitudinal mode, and when the electrodes were placed perpendicular to the applied force, the transverse mode. With regard to the magnitude of the extracted charge (Table 1, Figs. 3–6) there was a difference between the two brands of ceramics studied.

The experimental set-up was designed to minimize influence from generated triboelectricity (friction electricity). Each specimen in the present study was therefore fixed to the steel plate on the bottom of the electrically grounded aluminum can with copper foil tapes with conductive adhesive on both sides, whereas the upper end of the specimen was covered with electrically grounded copper foil tapes and a piece of tinfoil. These foils covered both the loaded area and a minor part of the surrounding area of the specimen. When the longitudinal mode was studied, the possibility cannot be completely excluded that friction electricity could have been generated on the surfaces of the copper foil tape and of the ceramic surfaces in the middle of the specimen when the specimens were loaded. However, the fairly linear relationship between the applied force and the extracted charge and the reproducibility of the magnitude of activity recorded when the force was cycled, as previously described, indicate that the contributions from generated friction electricity were negligible.

To minimize the effect of forces and movements reaching the polarity switch and the shielded output cable when the specimens were loaded, very thin spiral copper wires were used for the connections between the specimen and the polarity switch (Figs. 1 and 2). In

addition, a force of at least 100 N was applied on the specimen during each measurement. Moreover, to ensure that there were no spurious contributions from the experimental set-up itself which could influence the measurements, the steel rod inserted into the aluminum can was loaded with various forces without any ceramic specimen inside the can. Furthermore, a completely short-circuited and grounded dummy specimen was then placed in the aluminum can. Next to this specimen a regular specimen with copper foil tapes fixed on the surfaces opposite each other was mounted and connected to the charge amplifier as in the study of the transverse mode. Only the dummy specimen was thereafter mechanically loaded. In both these control tests forces up to 5500 N were used, and the loadings were carried out similarly to when the test specimens were studied, but no detectable effects on the oscilloscope display were recorded. Thus, the test arrangement was designed to minimize the influence from the environment and from generated friction electricity in particular. Yet charges could be extracted when the ceramic specimens were loaded, and there was a fairly linear relationship between the magnitude of the extracted charge and the force applied (Figs. 3–6). When the applied force was reduced to 100 N at the end of each loading cycle, the oscilloscope trace reverted to the base line of 100 N. Furthermore, when the electrode connections were shifted, by means of the polarity switch in the study of the transverse mode, the direction of the oscilloscope trace changed.

Moreover, to ensure that the charges extracted were not generated by the copper foil tape used, the tape that served as signal electrode when the longitudinal mode was studied was replaced by several copper wires (each wire diameter $\approx 15 \mu\text{m}$). The copper wires were placed between two pieces of the ceramic after the specimen had been separated into two equal parts by a cut parallel to its bottom surface and the upper part had been turned 180° as previously described. The other ends of the copper wires were connected, by soldering, to the signal cable of the charge amplifier by means of a single copper wire (diameter $\approx 15 \mu\text{m}$). The test was then carried out in the same manner as when the longitudinal mode was studied. The results showed that charges could also be extracted when this modified experimental set-up was used. When the connection to the signal cable was cut—that is, the single copper wire was cut—no detectable signal could be observed on the oscilloscope trace. Since there was a fairly linear relationship between the applied force and the extracted charge, it seems unlikely that the extracted charges were entirely caused by friction electricity.

To avoid any potential influence of friction electricity on the test, the transverse mode was also studied without using copper foil tapes. Two surfaces opposite each other were thereby covered with a thin layer of silver by means of evaporation. The spiral layer wires (diameter $\approx 15 \mu\text{m}$) were thereafter fixed to the silver-

coated surfaces by means of an electrically conductive epoxy adhesive (Circuit Works, Planned Products, Santa Cruz, Calif., USA). This epoxy adhesive covered $\approx 1 \text{ mm}^2$ of the ceramic surface. The test was thereafter carried out in the same manner as when the transverse mode was studied. Charges could also be determined with this modified test arrangement, and there was a fairly linear relationship between applied force and extracted charge.

To further ensure that the charges observed were generated on the surfaces of the specimen, two ceramic cylinders (Vita Celay Blanks, Vita Zahnfabrik) were pressed into holes that had been drilled in the steel plate fixed on the bottom of the aluminum can. In the upper end of these ceramic cylinders a hole had been drilled and a copper wire was pressed into each of them. Two silver plates were placed, at a distance of $\approx 1 \text{ mm}$ from the naked surfaces of the specimen, opposite each other on either side of the specimen and soldered to the copper wires. These silver plates measured $(10 \times 12 \times 0.3) \text{ mm}$ and were connected to the charge amplifier by means of copper wires (diameter $\approx 15 \mu\text{m}$) by soldering. The top surface of the specimen had been covered with grounded copper foil tapes and a piece of tinfoil, and the specimen had been fixed to the steel plate on the bottom of the aluminum can as in the previous experiments, whereas all the other surfaces of the specimen were naked. After this the specimen was loaded, as previously described, with a force cycled between 5500 N and 100 N. With this experimental set-up too charges could be demonstrated. To ensure that there were no spurious contributions from the experimental set-up itself, the specimen was replaced by completely short-circuited and grounded dummy specimens. When these dummy specimens were loaded, no charges could be observed. Moreover, when the humidity in the Faraday cage was increased, by means of a wad of cotton wool moistened with water, it was impossible to determine any charge. When, however, the wad of cotton wool was replaced by a drying agent (silica gel) for 30 min, charges could again be demonstrated. Thus, these control tests showed that charges could be extracted both without using copper foil tapes and also when the electrodes were placed at a distance of $\approx 1 \text{ mm}$ from the naked surfaces of the specimen. Consequently, it seems reasonable to assume that the charges extracted using the experimental set-up in the present study were created on the ceramic surfaces.

Another interesting finding in the present study was that the magnitude of the charge extracted from some of the Cerec Vitablocs specimens differed considerably from the other Cerec Vitablocs specimens studied. Conceivably, this difference might be the result of differences in composition, caused, for instance, by variations in the composition of the raw material and/or the direction in which the specimens had been cut. This hypothesis needs to be studied further.

In conclusion, on the basis of the results of the present

work it seems reasonable to assume that the charges extracted were created on the surfaces of the specimens. Furthermore, there are indications supporting the hypothesis that these charges were created by the direct piezoelectricity. However, further studies are necessary for decisive proof that true piezoelectricity occurs in the materials concerned.

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