

with MH were prepared by sectioning and polishing. Cross-sectional Vickers hardness of the 600  $\mu\text{m}$  thick enamel sections was measured across MH-affected and apparently sound areas and mean hardness values analysed by one-way ANOVA. Subsequently these sections were lapped to  $\sim 100 \mu\text{m}$  and assessed using polarizing microscopy (PM) and transverse microradiography (TMR). In specimens without post-eruptive breakdown (PEB) the visually sound surface layer was significantly harder (1.33–6.71 GPa) than the MH-affected enamel (0.07–6.14 GPa,  $p = 0.007$ ). Specimens with PEB displayed a narrow 're'-mineralised surface layer of  $\leq 5\%$  increase in porosity from normal enamel when viewed by PM, representing  $\sim 8\%$  greater mineralisation than adjacent MH enamel measured using TMR. Hardnesses of MH enamel (0.07–6.14 GPa) were significantly lower ( $p < 0.001$ ) than sound enamel (2.55–8.92 GPa). MH hardness was variable as was the position within the enamel layer of the most severely affected area. The previously reported transition zone was present when lesions were assessed in an occlusal-cervical direction whereas changes in hardness occurred suddenly beneath the surface layer. The results demonstrate that changes in enamel physical characteristics associated with MH are significant. Surface characteristics, particularly in areas of PEB, suggest the capacity for increasing the mineralisation (and hence properties) of MH enamel clinically in a manner similar to that demonstrated for early caries lesions.

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### Non-Contact Optical Profilometry for Detection of Surface Changes of Hydroxyapatite Discs during Acid Attack

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The aim of this study was to investigate the use of noncontact optical profilometry to determine changes in surface roughness during de- and remineralisation. The principle of noncontact optical profilometry is based on transmitting white light through a lens with built-in spectral aberration. The white light is divided into the full spectral field and each spectral component is focused at a slightly different height through a defined measuring range. When a reflecting object is placed within this range, only one particular colour frequency is reflected back from the surface. This information is then passed back to a spectrometer, which analyses the signal and converts it to a height measurement. The profilometer combines these measurements with the precise location of a moving X/Y linear table, creating three co-ordinates required for a 3-dimensional profile. The vertical confocal multiplexing sensor has a resolution of down to 0.01  $\mu\text{m}$ . In this study, a total of 5 HAP discs with a nominal 20% porosity (Plasma Biotol Ltd, Tideswell, UK) were used to measure the change in the surface roughness parameter  $R_a$  following exposure of the discs to an artificial caries demineralising solution (pH 4) for 38 h. Baseline Mean  $R_a$  was 1.78  $\mu\text{m}$ . The mean change in  $R_a$  was 0.12  $\mu\text{m}$ . Noncontact optical profilometry can be used to quan-

tify changes in the roughness parameters of the surface of HAP discs during exposure to acid, which may be a useful measure of the physical-chemical processes occurring during de- and remineralisation.

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### Proton (PIXE) Microprobe Investigations on Carious Changes in Human Teeth

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Since tooth material is essentially non-uniform, microprobe methods are most relevant for the study of spatial distribution of elements. The aim of this study was to assess the chemical signs of the initial phase of caries in human permanent molar teeth by micro-particle-induced X-ray emission (PIXE), using the proton microprobe  $\mu$ -PIXE. The collected elemental mappings were compared with backscattered electron or optical images, to relate the chemical information to the topography. Zones of initial and more advanced decay were visible. The advanced zone was depleted of mineral components. The zone of decay was surrounded by the endangered zone, 30  $\mu\text{m}$  wide, which was also clearly observed in the SEM images, and less clearly in the optical images. The occurrence of the endangered zone was confirmed by the chemical mappings. The wall-like zone was poor in calcium and phosphorus and enriched in magnesium, oxygen and carbon. In conclusion the application of PIXE microprobe gives the opportunity to observe changes in teeth leading to decay. The composition of the initial zone could suggest that there is an increase in the concentration of magnesium as the element initiating the formation of new apatite.

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### Photothermal Evaluation of Demineralization Kinetics of Human Enamel

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Photothermal radiometry was applied as a safe and non-destructive tool for monitoring the kinetics of human enamel demineralization. Teeth were treated sequentially with an acidified gel (pH 4.5) to simulate controlled mineral loss on the enamel surface. The experimental setup included a semiconductor laser (659 nm, 120 mW) as the source of the photothermal signal. Mod-

ulated laser-light-generated infrared blackbody radiation from teeth upon absorption and non-radiative energy conversion. The infrared flux emitted by the treated region of the tooth was monitored with an infrared detector after each treatment period. The measurements consisted of frequency scans with a laser beam size of 3 mm, in order to guarantee one-dimensionality of the generated photothermal field. The measured signal showed clear change even after 1 min of gel treatment, showing the excellent sensitivity of the method. Following the photothermal experiment, TMR analysis was performed in order to analyze the final structure of the enamel after demineralization. A theoretical model comprising coupled diffuse-photon-density-wave and thermal-wave boundary-value problems was developed to describe the photothermal phenomena in a tooth. From the fits of the measured frequency scans to the theory, thermal and optical properties of the enamel at different stages of demineralization were obtained, which allowed for quantitative monitoring of the demineralization kinetics. In conclusion, the developed method was shown to be a promising tool for non-invasive quantitative analysis of dental enamel demineralization.

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### A Comparison of Terahertz Pulsed Imaging with Surface Microhardness to Measure Mineral Changes in Enamel

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The aims of this study were to determine the ability of terahertz pulsed imaging (TPI) to measure mineral changes in enamel and to compare this technique with surface hardness (SH). 48 artificial lesions were formed in bovine enamel specimens using 0.1 M lactic acid (pH 5.0) containing 0.2% Carbopol C907 and 50% saturated with hydroxyapatite. The 20 day experimental protocol consisted of 4 1-min treatments with dentifrices containing either 10, 675, 1385 or 2700 ppm fluoride (1 part dentifrice:2 parts saliva), a 4 h/day acid challenge, and for the remaining time specimens were stored in a 50:50 pooled human/artificial saliva mixture. All specimens were imaged using a TPI imager 1000 (Teraview Ltd). Each sample was positioned at the focus of the beam and the optics raster scanning in the x-y plane to collect a grid of data points with line spacing of 50  $\mu\text{m}$ . SH was determined from an average of 4 indentations (50 g for 15s) at least 400  $\mu\text{m}$  from each other. For both techniques, baseline and 20 day measurements were made. The intensity change from baseline of the reflected air/enamel interface pulse and the % SH recovery were both dose-dependant. A statistically significant correlation was also observed between these 2 parameters (Pearson's  $r = 0.59$ ;  $p < 0.05$ ). This study has demonstrated that TPI is a useful technology to measure mineral changes in enamel and is sufficiently sensitive to discriminate between the levels of remineralisation produced by the different dentifrices.

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### A Comparison of Non-Destructive Techniques with Transverse Microradiography for the Measurement of Enamel Demineralisation

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Non-destructive alternatives to transverse microradiography (TMR) have often been evaluated using lesions with a limited range of mineral loss ( $\Delta Z$ ) and lesion depth (LD), or relatively shallow lesions. The aim was to evaluate quantitative light-induced fluorescence (QLF), optical coherence tomography (OCT) and terahertz pulsed imaging (TPI) with TMR as techniques for measuring demineralisation over a relatively wide range of  $\Delta Z$  and/or LD. Caries lesions were created by immersing bovine enamel blocks in lactic acid/methylcellulose gel, pH 4.6. These were removed after 7, 11 and 17 days to give lesions with a wide range of  $\Delta Z$  and LD. For QLF, the difference in fluorescence between sound and demineralised enamel ( $\Delta F$ ) was calculated. For OCT, plots of reflected signal intensity as a function of depth (ascans) were plotted; these were integrated (Ri values) and peak-width (PW) was measured. For TPI, samples at beam-focus were raster-scanned (data points 50  $\mu\text{m}$  apart) and LD calculated from time-of-flight data (ToF).  $\Delta Z$  and LD values were calculated using TMR. Qualitatively, lesions were a mixture of softening and sub-surface at 7 days and predominantly sub-surface at 11 and 17 days. Significant linear correlations (Pearson) were found between Ri,  $\Delta F$ ,  $LD_{TPI}$  and  $\Delta Z$  and increasing demineralisation time ( $< 0.05$ ). Significant linear correlations were found between;  $\Delta Z$  and  $\Delta F$ ;  $\Delta Z$  and Ri; LD and ToF, LD and PW. The findings demonstrate that OCT, QLF and TPI all have the ability to measure enamel demineralisation over a wider range of  $\Delta Z$  and LD than has often been reported.

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### TMR: Automatic Mineral Assessment Analysis of Wedge-Shaped Tooth Tissue Sections

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With TMR the accuracy of mineral loss (IML) and lesion depth (LD) strongly depends on the parallelism of tooth sections. Currently a rectangular area (sound patch) is positioned on the tooth section X-ray image where it is assumed to be sound. At each sound-patch pixel it is assumed that the volume percentage is of sound tooth tissue. Its average grey value is used to cal-