Measurement uncertainty associated with chromatic confocal profilometry for 3D surface texture characterization of natural human enamel

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ABSTRACT

Objective. To investigate the measurement performance of a chromatic confocal profilometer for quantification of surface texture of natural human enamel in vitro.

Methods. Contributions to the measurement uncertainty from all potential sources of measurement error using a chromatic confocal profilometer and surface metrology software were quantified using a series of surface metrology calibration artifacts and pre-worn enamel samples. The 3D surface texture analysis protocol was optimized across $0.04\,mm^2$ of natural and unpolished enamel undergoing dietary acid erosion (pH 3.2, titratable acidity 41.3 mmol OH/L).

Results. Flatness deviations due to the x, y stage mechanical movement were the major contribution to the measurement uncertainty; with maximum Sz flatness errors of $0.49\,\mu m$. Whereas measurement noise; non-linearity's in x, y, z and enamel sample dimensional instability contributed minimal errors. The measurement errors were propagated into an uncertainty budget following a Type B uncertainty evaluation in order to calculate the Standard Combined Uncertainty (u_c), which was $\pm 0.28\,\mu m$. Statistically significant increases in the median (IQR) roughness (Sa) of the polished samples occurred after 15 (+0.17 (0.13) μm), 30 (+0.12 (0.09) μm) and 45 (+0.18 (0.15) μm) min of erosion (P < 0.001 vs. baseline). In contrast, natural unpolished enamel samples revealed a statistically significant decrease in Sa roughness of −0.14 (0.34) μm only after 45 min erosion (P < 0.05 s vs. baseline).

Significance. The main contribution to measurement uncertainty using chromatic confocal profilometry was from flatness deviations however by optimizing measurement protocols the profilometer successfully characterized surface texture changes in enamel from erosive wear in vitro.

1. Introduction

Optical surface texture measurement and characterization of dental hard tissues are becoming increasingly used within dentistry as a method for detecting and quantifying early enamel damage resulting from common oral pathologies such as dental erosion [1,2]. When quantifying the micro-texture of enamel surface damage, the earliest signs of wear occur at the scale of an enamel prism. Accordingly, recent research into nanometer scale surface changes of polished human enamel using confocal laser microscopy concluded that optimal characterization of acid mediated surface texture changes

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requires surface metrology instrumentation with lateral resolution less than 2.5 μm [3]. Chromatic Confocal profilometry is industry standard for optical surface metrology and is specifically recommended by the ISO 25178 international standard for non-contact 3D metrology [4,5]. However, the lateral resolution of chromatic confocal profilometers is limited by the sensor spot diameter varying from up to 24 μm and also by lateral mechanical scanning which introduces measurement noise during the movement of the x, y stage [6]. In contrast the lateral resolution of confocal laser scanning microscopy is typically in the order of 100 nm and no mechanical scanning is required [7]. Therefore, as confocal chromatic profilometry is increasingly recommended for dental research into the topography and texture of natural and biological materials [2,8], there is a need to understand the measurement performance of surface metrology instrumentation operating at supra-micrometer level resolutions.

There are many potential sources of measurement error which can undermine certainty of measurement, including instrument bias, mechanical or optical changes due to aging, wear, or other kinds of drift, poor readability and environmental or electrical noise; as well as specimen issues such as dimensional instability, or other operator, process or environmental derived errors [9]. In the case of surface metrology for mineralized tissue and dental materials research applications, potential sources of error include those associated with the fundamental operating principles of the profilometer [10], as well as potential dimensional instability from enamel sample dehydration and rehydration during measurement [11]. One method of understanding the quality of a measurement system for a given application is to assess the uncertainty of measurement [9]. Uncertainty is a quantification of the doubt about the measurement result and is used in engineering industry to identify, quantify, and characterize each independent variable contributing errors in the measurement process, in order to evaluate and reduce these errors thus improving measurement quality.

The aim of this study was to investigate the measurement performance of a chromatic confocal profilometer for quantification of acid-mediated surface texture changes in human enamel. The objectives of this study were (a) to quantify the measurement uncertainty associated with chromatic confocal profilometry of human enamel undergoing erosive surface damage and (b) to optimize chromatic confocal profilometry for characterization of erosive surface texture changes in natural and polished enamel in vitro.

2.   Materials and methods

A chromatic confocal sensor (STIL OP350VM, France) mounted on a non-contact profilometer (Xyris 4000, Taicaan, Southampton, UK) operating with 3.5 μm lateral resolution and 10 nm vertical resolution was used under carefully controlled conditions throughout this study. As shown in Fig. 1, the optical principles of the sensor involved passing polychromatic white-light (a) through a chromatic lens (b) to generate a continuum of monochromatic light located on the optical axis. Samples surfaces (c), located within the optical z range below the sensor at a specified position on the x, y stage,

scattered the incident light beam back through the chromatic lens (b), via a beamsplitter (d) to a pinhole (e), which filtered the single reflected wavelength (λ) representing a set distance from the lens. A spectrometer (f) thus allocated the sample surface z position according to the detected wavelength of peak intensity (g). The stage then moved to the next x, y position in a raster pattern and thus the entire sample surface was scanned [7]. Resulting topography data were exported to surface metrology software (MountainsMap® V7.2; Digital Surf, Besançon, France), validated to ISO 3D surface metrology standards and all measurements were conducted by a single operator [4].

For the uncertainty analysis, potential sources of measurement error were identified following advice from dimensional metrologists at the UK's National Measurement Institute (National Physical Laboratory, Teddington, UK) and these were systematically investigated using calibration artifacts and the results were propagated using an uncertainty budget following good practice metrology guidelines [9,12,13]. Firstly, a calibrated optical flat (National Physical Laboratory, Teddington, UK) was used to quantify measurement noise added to the output signal occurring during the normal use of the instrument and flatness deviations which indicates the quality of the areal reference of the instrument. Three repeated 5 mm by 3 mm areas of the optical flat were scanned across five positions of the x, y stage; four in the peripheral corners and one in the central x, y position. Measurement noise was quantified using the maximum root mean square value of the scale limited surface (Sq) and flatness deviations were quantified using the measured maximum height of the scale limited surface (Sz) [4].

Lateral (x, y) linearity errors were quantified across 5 mm of a calibrated chrome-on-quartz linear scale (SC6 Lateral Scale, Microscopy Optical Dimensional Standard, National Physical Laboratory, UK) with 10 μm nominal line width and 100 μm nominal pitch [14]. The scale was positioned in x and y orientations and scanned three times per axis. Resulting 3D profile data were aligned parallel to x or y axes, following which a mean 2D profile was extracted and the raw 2D data were exported to spreadsheet software (Microsoft Excel 2010, Microsoft Office). The central point of each line on the lateral scale was identified thus allowing the mean (SD) difference between the nominal position of the center of each line on the lateral scale and the measured position of the center of each line on the lateral scale to be expressed across the x and y lateral axes and the maximum linearity error (μm) was calculated for each axis.

Vertical linearity errors were quantified along the z axis using calibrated glass 0.3 μm, 2.97 μm, 17 μm and 30 μm step height standards (Type A1 reference standard, Taylor Hobson Ltd., Leicester, United Kingdom). Each step height reference standard was placed onto the central position in the x, y stage and scanned at three positions across the z stage (high at +12.5 mm, middle and low at −12.5 mm), in order to quantify the contribution of non-linearity on the vertical scale [15]. The mean 3D step height was calculated by comparing the mean height of the central third of the bottom of the step with the mean height of the central third of the reference plane [16]. The mean (SD) differences between the nominal step height