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Makyoh-topography studies of the morphology of the cross sections of renal stones

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Abstract

Makyoh topography is applied to study the morphology of mechanically polished cross sections of renal stones. The results are compared to roughness and height profile data obtained by white-light interferometry, and to microstructure studies using optical and scanning electron microscopy. It is shown that Makyoh topography is able to furnish qualitative information on the surface roughness and height profile. Correlation with compositional data obtained by X-ray diffraction are established and discussed in terms of mechanical properties. Potential application for the study of the inner structure of the renal stones and implications for shock wave lithotripsy are discussed.

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1. Introduction

The studies of physical and chemical properties of urinary stones are important to understand their formation [1–3] and to assess their fragmentation behavior during shock wave lithotripsy (SWL) [4,5]. For the identification of the chemical composition, a host of analytical techniques are applied, such as X-ray diffraction, infrared and Raman spectroscopy. For morphological studies of both the outer surface and the inner structure, viewing by naked eye as well as optical and scanning electron microscopy are the most frequently applied techniques. To assess the SWL behavior, studies of acoustic and mechanical properties (elastic constants [4], microhardness and fracture toughness [6,7]) have been made. Because of the complex nature of the stone formation conditions, the chemical composition and the crystalline structure strongly varies from stone to stone and within a given stone. Therefore, no single method is able to provide complete

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information on the stones' properties, as it has been pointed out by many authors [2,3]. Consequently, applying novel techniques is desirable for a better understanding of stone structure and properties.

In this paper, we report for the first time the application of an optical method, Makyoh (or magic-mirror) topography [8,9] for the studies of the morphology of the cross sections of renal stones. Our results, obtained on a given stone, are compared to height profile and surface roughness data measured with white-light interferometry as well as to scanning electron and optical microscopy studies. Correlation with compositional data obtained by powder X-ray diffraction are established and discussed in terms of mechanical properties of the constituents. Application for the study of the inner structure of renal stones and implications for SWL are discussed.

2. Background on Makyoh topography

Makyoh topography is an optical method for the study of the flatness of mirror-like surfaces. The principle of the method is simple: the irregularities of the studied surface act as (local) concave or convex mirrors; therefore, a collimated light beam impinging on the surface produces a reflected image on a screen that is related somehow to the surface morphology (Fig. 1). The word Makyoh means 'magic mirror' in Japanese and refers to an ancient sacred mirror originating from the Far-East: such mirrors were hung on Buddhist temple walls and reflected an image corresponding to their back relief pattern onto a distant wall when they were shone by the Sun [10]. The image is due to the small irregularities of the front surface caused by the back pattern during machining of the mirror. Nowadays, Makyoh topography is mainly applied in microelectronic technology to assess the flatness of semiconductor wafers used in the circuit fabrication processes [8,9].

A comprehensive geometrical optical model of Makyoh imaging has been presented in Ref. [11]. The determining parameter of the imaging is the screen-to-sample distance, L. The position of a given surface point's image is determined by the surface slope and L, while the image point's brightness by the surface point's reflectivity, the local curvatures of the surface and L. Surface roughness causes a convolution (blurring) of the image with a weight function proportional to L. At $L = 0$ we get the reflectivity image: the imaging plane is the sample surface, and image contrast results solely from the reflectivity differences of the surface. With increasing $|L|$, the image becomes increasingly dominated by the contrast due to surface morphology. Usually, taking more images with different L settings is necessary, especially if the surface reflectivity is not uniform.

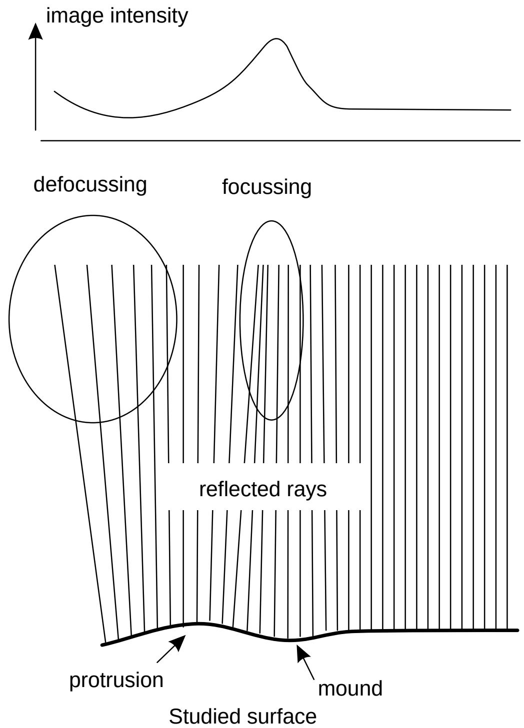

Fig. 1. Scheme of Makyoh-topography imaging.

3. Experimental technique

3.1. The stone, preparation and compositional analysis

The stone was obtained from a 77 years old male after open surgery. The cross section was prepared as follows. The stone was first rinsed in running distilled water for a few minutes then air-dried. The sample was then cut into two halves with a diamond wheel and was lapped with 5-μm SiC powder, then polished successively with 1.0- and 0.3-μm Al_2O_3 powder on polishing cloths with the final polishing using 0.1-μm SiO_2 suspension. Finally, the sample was cleaned ultrasonically in distilled water, then air-dried.

Fig. 2 shows the in-focus image of the section of the stone taken in diffuse laboratory light by the Makyoh instrument. A radial line and its characteristic points corresponding to the well-distinguishable regions are marked for further reference; the interferometric and microscopic studies were performed along this line. The stone exhibits a complex structure of the centrally situated porous core region with two cores and concentric laminated peripheral layers. The composition of the stone regions was determined by powder X-ray diffraction analysis of the material removed from selected locations of the stone, marked in Fig. 2. An X-ray equipment (type Philips X'pert, working regime, 40 kV and 30 mA) with Cu Kα radiation ($\lambda = 0.154$ nm) through a Ni filter for the Kβ radiation was used. A computer code Crystallographica Search-Match from Oxford Cryosystems was used for the identification of X-ray diffraction spectra, and the determination of the compositional ratio for the mixed-composition stone layers (strata) was carried out by the X'pert Plus Philips code. Table 1 summarizes of the results obtained by the X-ray studies. The chemical composition of the stone is typical for renal stones [1,2]. The similar visual character and the identical composition of the two opposing points of strata D and E (D1 and D2, and E1 and E2, respectively) suggests that the composition is more or less uniform within a given stratum, that is, any radial line well represents the whole stone.

3.2. Makyoh-topography setup, interferometric and microscopic studies

Our Makyoh setup [9,11] uses a pigtailed LED (wavelength, 820 nm) for illumination collimated by an 80-mm diameter, 500-mm focal length lens and an 800 × 600 pixel resolution CCD camera for image observation (Fig. 3). The camera video output is fed to an 8-bit frame grabber card inserted into a PC with appropriate image-processing software. This setup is equivalent to the original arrangement with a simple screen [12]; in our setup, the equivalent L can be varied from 500

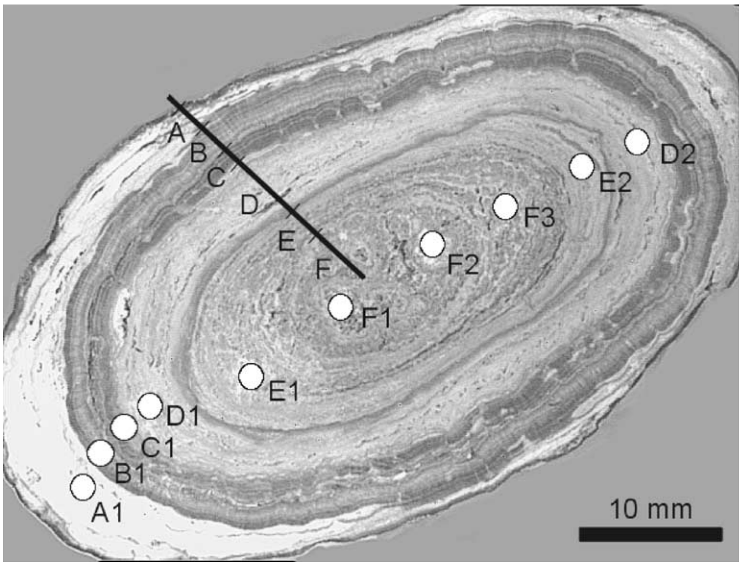

Fig. 2. In-focus image of the stone. The marks refer to locations of the studies; see text.

Table 1
The chemical composition of the strata of the studied stone in weight %

Position in image	Composition (mineralogical name and chemical formula)
A1	Newberyite ($MgHPO_4 \cdot 3H_2O$)
B1	Whewellite ($CaC_2O_4 \cdot H_2O$)
C1	Whewellite ($CaC_2O_4 \cdot H_2O$)
D1	Ammonium hydrogen urate($C_5H_7N_5O_3$) (24.4%) + newberyite ($MgHPO_4 \cdot 3H_2O$) (75.6%)
D2	Ammonium hydrogen urate ($C_5H_7N_5O_3$) (24.4%) + newberyite ($MgHPO_4 \cdot 3H_2O$) (75.6%)
E1	Uric acid ($C_5H_4N_4O_3$)
E2	Uric acid ($C_5H_4N_4O_3$)
F1	Uric acid ($C_5H_4N_4O_3$) (27.3%) + whewellite ($CaC_2O_4 \cdot H_2O$) (72.7%)
F2	Uric acid ($C_5H_4N_4O_3$)
F3	Uric acid ($C_5H_4N_4O_3$) (20.3%) + whewellite ($CaC_2O_4 \cdot H_2O$)(79.7%)

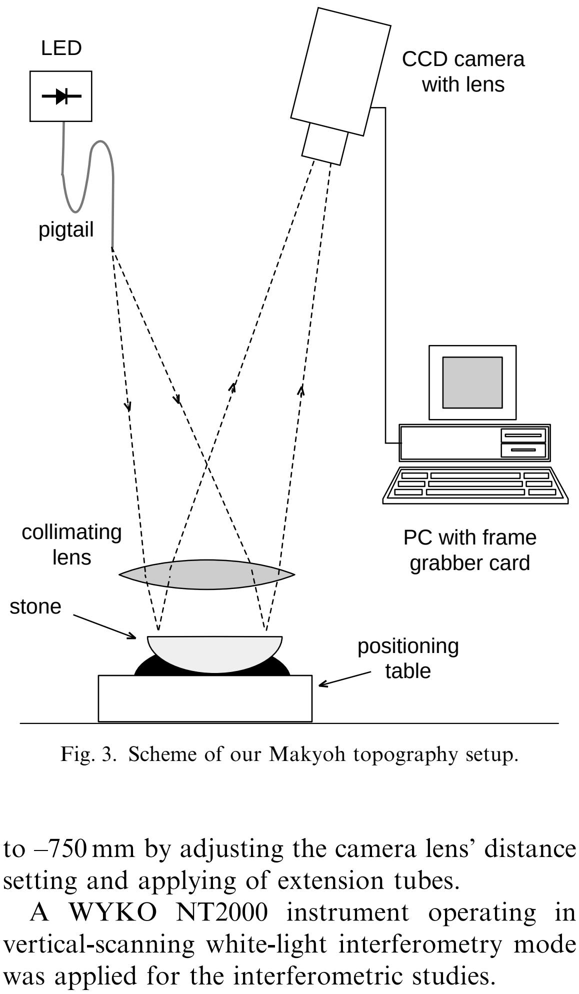

Fig. 3. Scheme of our Makyoh topography setup.

to –750 mm by adjusting the camera lens' distance setting and applying of extension tubes.

A WYKO NT2000 instrument operating in vertical-scanning white-light interferometry mode was applied for the interferometric studies.

The scanning microscopy studies were made using a Jeol JSM-25 scanning electron microscope (SEM) in secondary electron image mode. A low accelerating voltage of 2.5 keV was applied to avoid charging effects. In addition, optical microscopy studies were performed using bright-field illumination.

4. Results

Fig. 4 shows the Makyoh images of the stone taken at different L settings (the whole image is shown for $L = 0$ only; for the other L settings, only the upper left quarter is shown to save space). The light intensity was set just below the onset of saturation of the CCD sensor; the LED power was 120 μW in this case. The position of the stone and the magnification of the instrument is the same for all L settings as for Fig. 2. The $L = 0$ picture is highly structured reflecting the inner structure of the stone (cf. Fig. 2). The strongly reflecting regions are B, C and D while the others' images (A, E and F) are rather dim. The low image intensity of these areas is not due to low surface reflection since they reflect well in scattered light (cf. Fig. 2), rather to the high roughness: the reflected rays scatter out of the field of view of the Makyoh instrument. The high roughness indicates a porous structure of the stone region. The image of stratum C is sharper and more uniform than that of B; this hints to the higher surface roughness of stratum B. Increasing $|L|$ increases the degree of blurring of the images, while the individual strata remain distinguishable; at $|L| > \sim 150$ mm, the

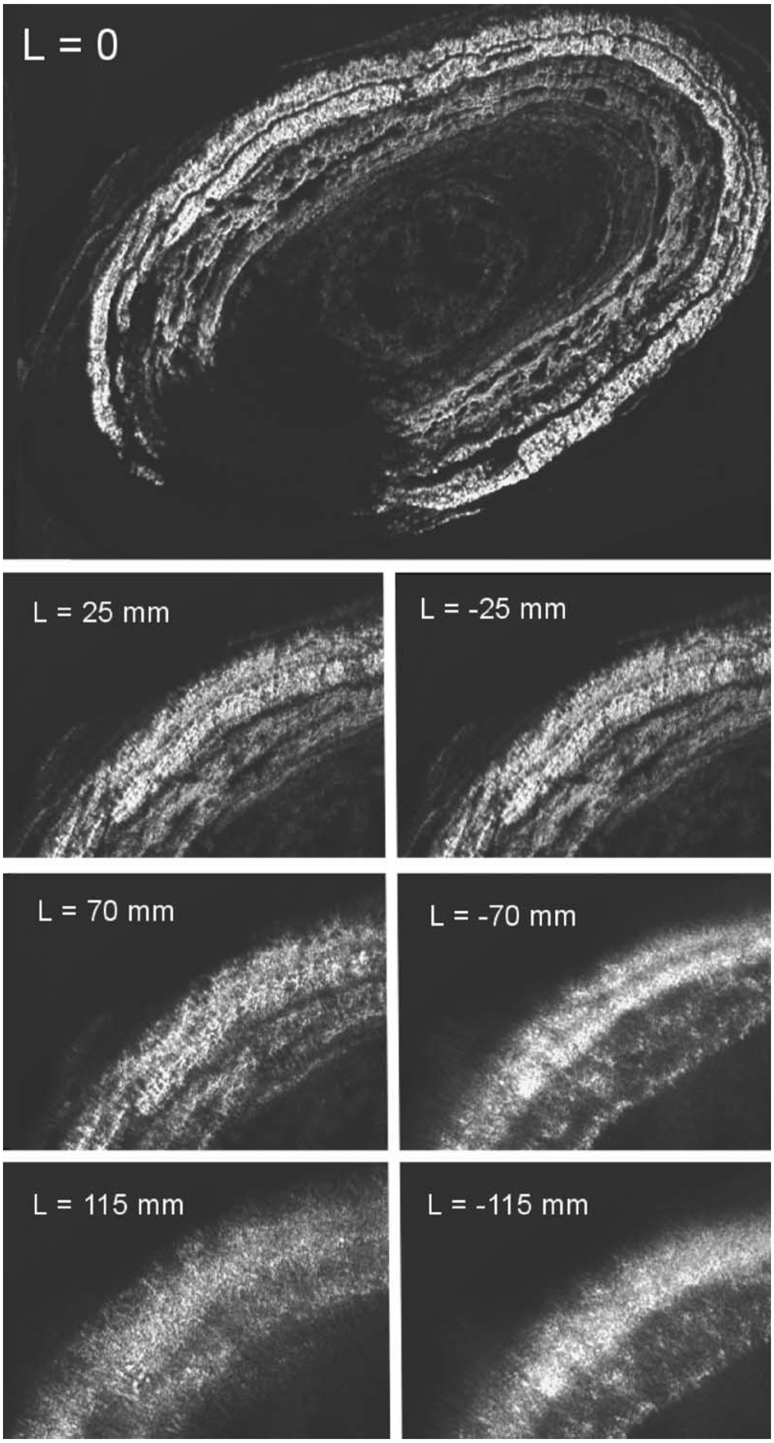

Fig. 4. Makyoh images of the renal stone at different L parameters.

images become totally "hazy" with no well-defined structure.

The images of strata B and C move away from each other if L changes in the positive direction. This indicates that they incline away from each other (like a tent).

The image of stratum D is inhomogeneous, both in radial direction and along the stratum. This indicates medium-scale ($\sim$mm) surface undulations. Moreover, the outer bright line moves towards the center when L changes in the positive direction. This indicates sloping of the outer part toward the center of the stone.

The results of the interferometric measurements of the height profile and the Rq (RMS) roughness [13] values are shown in Fig. 5. The Rq values were evaluated in a $0.3\,\text{mm} \times 3.5\,\text{mm}$ (40×480 pixel) box perpendicular to the line marked in Fig. 2 in the line's characteristic points. The discontinuities of the profile curves are due to deep scratches or voids. The roughness data correspond to the results of the Makyoh study: the less rough regions' (B, C and D) images are more defined and of higher intensity. In addition, in most cases the deeper regions of the profile (A, E and F) possess higher roughness. The height profile also confirms the conclusions drawn form the Makyoh images: the stratum B is indeed rougher than C, and their tent-like inclination is also confirmed. As for stratum D, the sloping of its outer part is again confirmed.

The morphological observations gained from the interferometric measurements are confirmed also by the SEM data as well (Fig. 6): region A shows structured, laminar features while regions B and C have structureless smooth surface. Stratum D has a long-range structure shown also by the Makyoh image (Fig. 4), while regions E and F exhibit porous surface. Optical microscopy confirmed the information gained from the SEM studies.

Note that the microscopy images are unable to provide information on the large-scale morphological features, namely, the tent-like inclination of strata B and C, and the large-scale roughness of stratum D; in this respect, Makyoh topography has a clear advantage over any microscopy methods.

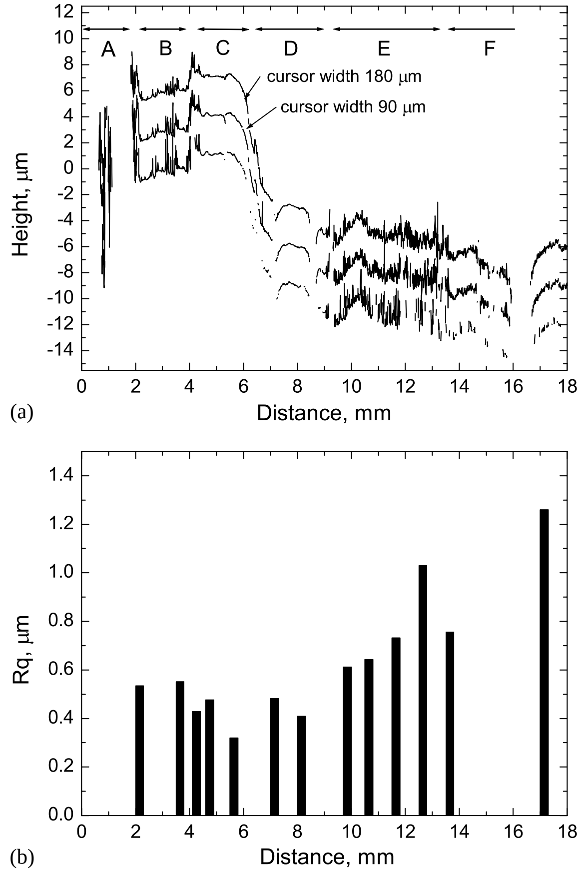

Fig. 5. (a) Surface profile of the stone (the curves with different cursor widths are displaced vertically for clarity) and (b) the Rq roughness data as determined by white-light interferometry along the line indicated in Fig. 2. The widths of the columns represent the size of the boxes in which the roughness figures were evaluated. For a clearer observation of the profile, curves of the same profile in which the profile was averaged over 90 and 180 μm widths are also included.

5. Discussion

The morphology obtained after mechanical polishing is the result of both the stone's inner properties (composition and crystallite structure, mechanical stress) and of the polishing conditions. If the polishing process is well controlled, correlation with the results of other (analytic and morphological) methods might facilitate the assigning of definite Makyoh 'fingerprint' patterns to certain compositions and growth habits.

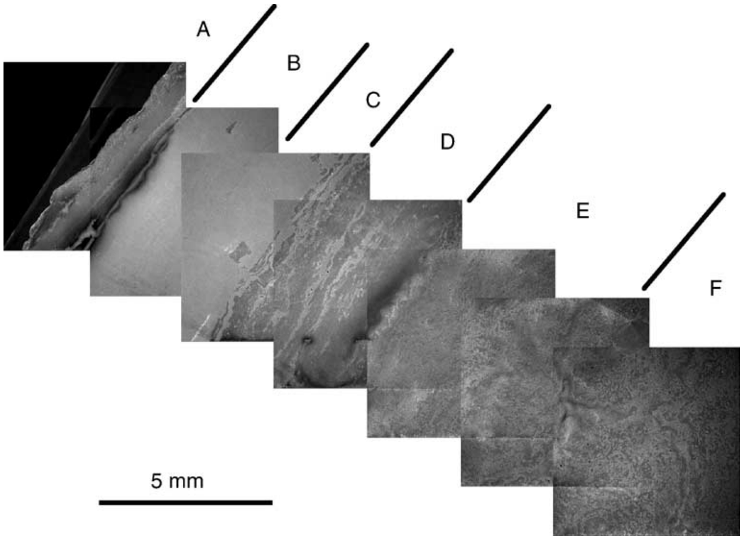

Fig. 6. A composite SEM micrograph of the stone along the radial line marked in Fig. 2.

The correlation between surface roughness and the resistance to fragmentation during SWL has been pointed out [14]. A more direct correspondence may also be formulated, since both the fragmentation process [5] and the polishing fritter away the material. Indeed, in our case higher roughness accompanies the deep regions of the profile (strata A, E and F), which means lower resistance of material to mechanical polish removal (the material is softer), and presumably, to SWL.

A correlation between composition and roughness can also be given since whewellite has considerably greater fracture toughness and microhardness than either uric acid [7] or uric acid mixed with whewellite [6]. It is also a common clinical experience that whewellite is more resistant to fragmentation during SWL than uric acid. This is consistent with our study since we have observed smaller roughness and higher resistance to polish removal of strata B and C, the whewellite regions of the stone.

Although the size of studied surface area can be changed easily [15], it is evident from our studies that the main advantage of Makyoh is the unique ability of studying large surface areas, thus complementing higher-resolution (and, consequently, small-area) methods such as SEM or optical microscopy. The resolution of Makyoh topography is in the order of $\sim 100\,\mu m$ assuming typical stone size of 1–3 cm and a size of the stone's image that equals 500 pixels of the camera's CCD chip.

Finally, it is important to note that Makyoh studies require the surface to be reflecting, that is, not too rough. A useful guide that if the surface looks 'shiny' by naked eye, it is evaluable by Makyoh. Standard polishing techniques yield a satisfactory quality surface.

6. Conclusions

In conclusion, we have demonstrated that Makyoh topography is able to yield qualitative and reproducible information on the inner structure of renal stones. Our results show that Makyoh topography has a potential to be a complementary method in the qualitative or semi-quantitative studies of the morphology of renal stones. It is important to recapitulate that Makyoh topography, unlike microscopy methods, has an ability to reveal long-range surface undulations and slope changes, which relates to the regional differences in mechanical properties of the stone. The method is particularly suitable to study inhomogeneities within a given stone, select regions for other (small-area) studies or to compare different stones. We finally note that Makyoh is a low-cost and fast technique and it requires no special adjustments.

Acknowledgements

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