Dry friction between laser-patterned surfaces: Role of alignment, structural wavelength and surface chemistry

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Abstract

The ability to tune friction by tailoring surface topographies at micron length scales and by changing the relative orientation of crystallites at the atomic scale is well established. Here, we investigate if the two concepts combine, i.e., we explore if the relative orientation of surfaces affects dry friction between laser-textured surfaces. Line-like laser patterns with varying structural wavelengths or periodicities (5, 9 and 18 μ m) have been fabricated on austenitic stainless steel substrates and on tribometer testing balls made of 100Cr6. Dry sliding tests for the mentioned wavelengths and 0°/90° relative alignments between both laser-patterned contact surfaces were done. We observe that the patterning reduces friction after running in. The reduction increases with decreasing wavelength and also depends sensitively on the relative alignment and the chemistry of the sliding surfaces. Our results highlight the possibility to create bespoke contacting surface geometries leading to tuneable frictional properties.

Key Words: Laser Interference Patterning, Dry Friction, Interlocking

Introduction

Most machines contain components with loaded solid surfaces that rub together. The interaction of such surfaces produces friction and also results in mechanical damage to them. Thus, tribological phenomena play a decisive role in diverse systems. For many years, researchers have sought to alleviate these problems and to understand their origin. The early work of Bowden and Tabor [1] showed that one influential aspect on friction is the interaction of asperities. They can be welded together and released by applying a critical shear stress τ_c . However, studies at different spatial scales revealed that tremendously small shear stresses between atomically flat surfaces are possible when their surface corrugation does not match [2]. Such a state of nearly vanishing friction was introduced by the pioneering work of Hirano and Shinjo [2] for incommensurate i.e. non-matching contacts and confirmed experimentally by Dienwiebel et al. on the nanometre scale [3]. Recently, Sondhauß et al. have demonstrated the influence of a mesoscopically patterned silicon surface on the frictional response. They used a silicon substrate which was patterned by the use of a focused ion beam microscope (FIB) and as a counterbody titanium spheres (without pattern) with different radii being attached to a slightly modified AFM cantilever. It could be shown that the effective coefficient of friction has characteristic maxima depending on the groove widths in the silicon substrate and the corresponding radius of the titanium spheres [4].

The ability to control and modify frictional forces on different scales bears promise for the design of miniaturised systems, positioning devices and bearings [5]. There are two major threads to manipulate friction by modifying the surface materials or texturing the relevant surfaces [6, 7]. In particular, laser surface texturing (LST), introduced by the Etsion group for the texturing of mechanical seals, proved to be a promising technique for dry and lubricated contacts [8-11]. One possible approach to LST is laser interference metallurgy. It is based on the interference of laser beams from a solid-state laser, which creates well-defined surface topographies on the micron scale [12-15]. Moreover, due to a periodic laser intensity distribution and significant heating /cooling rates of up to 10^{10} K/s [16], metallurgical effects like melting, resolidification and the formation of intermetallic phases can be induced [17-18]. In the present work, we apply the aforementioned technique to pattern both interacting surfaces, rather than just one, to control the involved contact geometries under dry sliding conditions. Using two-beam interference, line-like patterns with varying periodicity of the laser intensity maxima were fabricated. Furthermore, the as-patterned contacting bodies were scrutinised with regard to their ability to interlock depending on the structural wavelength and the relative alignment. In addition, microstructural (Xray diffraction) and chemical analysis (Raman spectroscopy) were applied to

study the laser treated specimens. Finally, experimental results of the frictional response will be presented and discussed.

Experimental Procedure

A commercially available austenitic stainless steel (1.4301) was used for the laser experiments. The lateral dimensions of the nominally flat specimens are 20 x 20 mm². The samples were delivered with a highly polished mirror-like surface finish having a root mean square roughness of about 30 nm. A pulsed Nd: YAG laser (*Spectra Physics, Quanta Ray PRO 290*) with a pulse duration of 10 ns was used for the laser structuring. The primary laser beam was split into two sub-beams to interfere with each other on the sample surface resulting in a line-like interference pattern (figure 1).

The intensity of each beam can be controlled using suitable beam splitters. The laser fluence was set to 400 mJ/cm² for all specimens. Due to the absorption characteristics of the used material, the third harmonic at 355 nm was selected. The periodicity (line-spacing) was set to be about 5, 9 and 18 μ m. All samples were irradiated at normal atmospheric conditions in air using one single laser pulse. Further details of the experimental setup have already been published elsewhere [13].

The topography was measured by a *Zygo New View 100* white light interferometer (WLI) equipped with a 3-D imaging surface structure analyser.

The surface morphology was imaged by scanning electron microscopy (SEM) (*FEI, Strata DB 235*). In addition, a focused ion beam microscope (FIB) (*FEI, Strata DB 235*) was selected to prepare cross-sections in order to investigate the microstructure.

XRD phase analysis measurements using grazing incidence geometry (GI-XRD) were carried out in order to achieve statistically relevant phase data of a significantly large specimen volume. For this purpose, a seven-axes diffractometer (*PANalytical X'PERT MPD Pro*) with a copper x-ray tube was used.

Raman spectroscopy was performed with a *LabRAM ARAMIS* instrument from *HORIBA* using a 532 nm laser beam without filter. The Raman microscope uses a back-scattering geometry, where the incident beam is linearly polarised and the spectral detection unpolarised. The optical elements used in this analysis allowed a laser spot size of 5 μ m (full width at half maximum) and depth penetration of

~ 1 μ m, while the spectra precision was around 5 cm⁻¹.

The tribological tests were performed with a nanotribometer using a ball on disk configuration in a linear reciprocating sliding mode (*CSM Instruments*) with a stroke length of 0.6 mm. The tribometer is based on a stiff cantilever, which acts as a frictionless force transducer in both vertical and horizontal directions. The static partner is loaded onto the substrate with a precisely known force using piezo-actuation. The normal load and the friction force are determined during the experiment by measuring the deflection of this elastic arm in both horizontal and vertical planes with two high precision fiber optical displacement sensors. Through a feedback loop, the piezo-actuation maintains the normal load independent of any surface irregularities. The normal force was set to 1 mN and the linear sliding speed to 1 mm/s. The deviation of the aforementioned normal load was less than 10 % related to the reference value in our experiments. The counterbody consisted of a typical 100Cr6 bearing steel ball with a diameter of 3 mm. Temperature and relative humidity were kept constant at $20 \pm 2^{\circ}$ C and 45 ± 5 %.

Results and Discussion

Topographic analysis

Figure 2 shows a typical line-pattern on a stainless steel substrate with a structural wavelength of around 9 μ m. The pattern, resulting from two interfering beams, is uniformly distributed and largely homogeneous.

The observed line-pattern is a result of a temperature gradient between positions of maximum and minimum laser intensity. It induces a surface tension gradient, which shifts molten material away from the hot regions. The shifted material resolidifies in the intensity minima. In addition to optical microscopy, WLI measurements were performed in order to analyse the topography of the resulting pattern on the substrate and ball surface (see figure 3).

For a geometrical interlocking of the two patterned surfaces, the groove and asperity widths of the contact surfaces are essential. Figure 3 shows cross-section plots of a patterned steel substrate and a tribometer ball measured by WLI. The laser periodicity in both cases was set to 18 μ m with a depth of around 1 μ m. The analyzed groove widths of the profiles presented in figure 3 range from 12 to 14 μ m in the upper part and 7 to 9 μ m in the lower part of the structured substrate.

The asperity widths for both surfaces range between 3 and 10 μ m. Therefore, an interlocking of the structures based upon the topographic analysis is generally possible.

Additionally, the line-patterns with varying periodicity were studied with regard to surface profile parameters and compared to each other.

The slope roughness S_{rms} , the skewness R_{sk} , the kurtosis R_{ku} and the Swedish height H were determined by WLI. In particular, the Swedish height represents the distance between two reference lines where the upper reference line covers 5% of the data and the lower one 90%. As a result, this value is less sensitive to surface artifacts such as spikes which may appear during WLI measurements [19]. Figure 4 depicts the surface profile plots for the three different periodicities. The height deviations for a line-spacing of 5 µm are more pronounced than for the other periodicities. When decreasing the periodicity, the lateral thermal flow becomes more important thus setting a lower physical limit, which is similar to the thermal diffusion length for most metals [20]. If laser patterns with periodicities in the range of the thermal diffusion length are created, the thermal gradient between the positions of maximum and minimum laser intensity will be reduced.

Consequently, the specific metal will be molten in both positions. According to Lasagni et al., the minimum achievable structural wavelength in stainless steel is about 1.3 µm [20]. Table 1 summarises the mentioned surface parameters. The average values are based on ten measurements of the respective parameters. The rms value of the reference sample is relatively low compared to the laserpatterned surfaces. This is due to the mirror-like surface finish of the specimens. The Swedish height H shows a maximum value of around 1.6 µm for a periodicity of 18 µm. There is a direct correlation between the structural depth and the used structural wavelength, already studied by Lasagni [21]. Usually, this depth increases for larger structural wavelengths. This is attributed to the aforementioned thermal gradient, which is generally smaller for lower periodicities. Furthermore, the reference specimen has a negative skewness indicating good bearing properties. In contrast, all laser-patterned surfaces show positive values due to the reduced bearing capability. The highest positive value can be found for the sample with the largest structural wavelength. Apart from the reference, the 9 μ m pattern has the lowest positive R_{sk} value. A comparison with the corresponding cross-section profile plot reveals a very homogeneous laser-pattern. During the laser induced melting process, the material is shifted away from the intensity maximum towards the minimum positions. In case of larger wavelengths, the distance between the maximum and minimum positions is expanded and so the distance in between cannot be bridged by the molten material. As a consequence, additional topographic maxima may appear with different heights.

The slope rms, S_{rms} , is listed for the various specimens. This parameter is particularly interesting because it represents the square-root of the second order spectral moment, which can be found in literature as $(m_2)^{1/2}$, and is roughly inversely proportional to the real area of contact [22]. As expected for similar structure depths, S_{rms} is highest for the smaller structural wavelength.

Microstructural and mechanical characterisation

The pristine steel surface is shown in figure 5. Some polishing artifacts are still visible. Despite the polishing marks, a coarse-grained microstructure is revealed by using ion channeling contrast in a SEM/FIB work station. A direct comparison with a laser treated sample surface does not disclose any considerable difference in the positions of maximum laser intensity (see figure 6). Due to the relatively coarse grains, the resolidifying microstructure is similar to the original one. This can be seen more clearly in a corresponding FIB cross-section (figure 7). Even in the positions of maximum laser intensity, the grain structure is not affected in an apparent way. Austenitic stainless steels are generally composed of the metastable γ -phase [23]. Thermal simulations were performed for the different structural wavelengths which have not shown any strong difference concerning the achievable temperatures in the positions of maximum and minimum intensity or the respective heating/cooling rates. Accordingly, the maximum temperature was approximately 1900 K (in the maximum position) which is clearly above the corresponding melting range of the used austenitic steel (T_m is between 1693 and 1747K [24]).

In this context, X-ray diffraction was applied to study the effects of phase formation in the chosen steel before and after laser patterning. The corresponding diffractogram in figure 8 could be ambiguously identified and assigned to the austenitic phase in both cases. The structural wavelength of the laser-patterned sample was 18 μ m in this case. Similar XRD results were found for a periodicity of 5 and 9 μ m. Furthermore, nanoindentation experiments were done in order to measure the hardness in the positions of maximum laser intensity and in the unpatterned reference regions. A Berkovich (three-sided pyramidal tip, 142.3°) diamond indenter was carefully calibrated for its area function on a standard testing material. The loading profile included three segments: loading to a 300 µN peak load within 5 s; holding at the peak load for 15 s; unloading back to zero loads in 5 s. No considerable thermal drift during the measurements was observed. The loading rate for all indents was set to 60 µN/s. Figure 9 shows the results of the hardness measurement for the polished reference and the laser patterned surface. Here, only laser intensity maxima, representing the topographic minima could be indented. Due to the melting process, the laser intensity minima are quite inhomogeneous and too narrow to be indented (see figure 6). Despite some data scatter, most of the values are similar and thus consistent with the previous SEM images. The average hardness value and standard deviation for the reference is 2.24 ± 0.55 GPa and for the patterned specimen 2.45 ± 0.62 GPa. In conclusion, no severe micro-mechanical changes could be detected by the methods used in this work. However, further high-resolution techniques will be necessary to study the microstructure in more detail.

Surface chemistry

Raman analysis was carried out on the structured samples in order to study the chemical species present in different topographic zones. In order to obtain representative and confident information, the analysis was performed ten times on each topographical zone identified in figure 6. As expected, similar species were found in the non-irradiated and irradiated areas (see figure 10). The low-frequency peaks located at around 210 cm⁻¹ and 270 cm⁻¹ correspond to vibrations of Fe₂O₃ observed in Fe-Cr-Ni alloys [25] and steel [26], whereas the peak located at ~380 cm⁻¹ was observed in (Fe, Cr)O₃ [27]. Moreover, the modes with wave numbers at around 590 cm⁻¹ and 640 cm⁻¹ have been observed in several materials containing FeO, Fe₃O₄ and Fe₂O₃ [28-31].

It is worth to note the change of the relative intensities of the peaks located at around 590 cm⁻¹ and 640 cm⁻¹. As expected, similar intensities with respect to the background are observed in the non-irradiated and minimum irradiated samples, whereas the intensity of the peak located at ~ 640 cm⁻¹ increased in the radiation maxima zones. Since the exact vibration of this mode is not clear within the

literature [25-31], the interpretation of this observation is not straightforward. Besides FeO, this mode may have contributions from magnetite (Fe_3O_4), maghemite ($-Fe_2O_3$) and hematite ($-Fe_2O_3$), due to phase transformations induced by high-temperature processes [30-31].

Moreover, as shown in figure 10, the peaks at ~ 270 cm⁻¹ and ~ 640 cm⁻¹ are shifted between the spectra of the 99.99 % pure iron and the radiation maxima zone, and the intensity/background ratio of the mode associated with (Fe, Cr)O₃ species decreased in the maximum radiation zone. The synergy of these evidences strongly indicates a modification of the oxidation state of the surface after laser patterning.

Tribological results

Finally, the as-patterned surfaces were analyzed in linear reciprocating sliding tests. To ensure stochastic significance and reproducibility, all tests were repeated at least ten times. In order to get a better understanding of the tribological kinematics and the relative alignment of the contacting bodies, figures 11 (a) and (b) depict a 3-D view of the structured steel sample and the tribometer ball. The sliding velocity was kept constant at 1 mm/s. The labeling 0°/90° denotes the angle between the structures and the black arrow indicating the sliding direction in our experiments. In addition, the friction coefficient μ as a function of sliding time for two different periodicities is shown in figures 11 (c) and (d). The displayed curves in figures 11 (c) and (d) are averaged plots of the friction coefficient of forward and backward strokes. After having exceeded the initial static friction, µ for the polished reference surface is larger than that of the patterned specimens independent of the relative alignment $(0^{\circ}/90^{\circ})$ between the rubbing partners. A closer look at the orientation of the surfaces reveals that the results for the 5 and 18 μ m structural wavelengths demonstrate a lower μ for a 90° alignment although the (initial) surface chemistry and the microstructural properties are obviously independent of the relative orientation. Therefore, it can be concluded that this observation is consistent with a structural incompatibility compared to the 0° alignment and finally to a reduced contact area [32]. In the case of a 0° alignment, the initial value of μ after the laser treatment ($\mu \sim 0.4$) deviates from the reference value ($\mu \sim 0.27$) of the pristine surface independent of the chosen structural wavelength. This might be explained by a potential interlocking at 0°, a different surface chemistry and microstructural situation after the laser treatment (e.g.

higher defect density). However, while interlocking automatically increases static friction, it does not necessarily explain an increased kinetic friction.

A closer look at the resulting wear scars for the two different sliding directions $(0^{\circ}/90^{\circ})$ is shown in figure 12. As expected in the case of 0° alignment, the wear track is more pronounced whereas for an angle of 90° only a slight wear scar can be identified.

A comparison of figures 11 (c) and (d) further implies a strong effect of the different structural wavelengths.

The temporal evolution of μ for the 0° orientation is demonstrated in figure 13 for three different wavelengths (5, 9 and 18 μ m). The coefficients of friction of all the laser -patterned specimens are initially higher than the value for the pristine unpatterned surface. The reference value starts at about 0.27 and increases with sliding time. This increase of μ over the sliding time can be divided into two parts with differing slopes indicating a change in the friction mechanism. Raman spectroscopy in combination with focused ion beam cross-sections has revealed the existence of a native oxide layer in the reference material. The typical oxide layer thickness is in the range of ~ 5 nm. Measurements of the oxide layer after the laser treatment by FIB cross-sections have demonstrated much thicker oxide layers with a thickness of approximately 26 nm in the intensity maxima (topographic minima). Because of the inhomogeneous structure of the intensity minima (topographic maxima), no reliable thickness of the oxide layer could be determined within the cross-section.

It is known from literature that the steel-steel friction coefficient is smaller with oxide layers on top (typically 0.27) than without under dry and clean conditions ($\mu \sim 0.80$) [33]. The friction coefficient of the reference in the beginning is in good agreement with the reported μ of 0.27.

The initial values of μ of the laser patterned surfaces are approximately 30 % higher compared to the reference surface for a 0° orientation. This may be attributed to an interlocking effect as well as a modified chemical and microstructural state of the laser irradiated samples in which the composition of the oxide layers differs from the pristine material surface and the defect density (e.g. dislocations) may be increased. Subsequent to the initial situation, μ of the laser-patterned surfaces decreases independent of the used periodicity. An explanation of this decline after some run-in could be that the induced patterns

gradually degrade, which can reduce the interlocking of asperities. The local properties of the patterned specimens become similar to those of the reference. After the sudden drop of μ , an increase can be observed which may be linked to further flattening of the laser structures thus causing a larger real contact area and a changed surface chemistry.

In figure 14, a direct comparison of the laser-induced surface profile is presented before and after sliding. The profile amplitude after sliding is visibly reduced and the profile looks more flattened after the sliding deformation.

Furthermore, after a sliding time of around 90 s, a deviating slope for all the specimens is visible. During sliding, oxide wear particles are created and shifted to the reversal points of the slider thus accumulating at the end of the wear track as indicated by light microscopy. As a result, the friction regime is changed because the oxide layer is gradually removed and the friction coefficient approaches the reported value of 0.8 for the steel/steel contact without oxide layer. While the surface chemistry and surface topography certainly play important roles concerning the initial values and various slopes within the friction curves but cannot explain the significant differences in μ for 0° and 90° orientation. One difference between the different orientations is their relative contact areas. They would yield different total friction assuming local friction-load relationships of the form $F_s = \sigma_0 A_{real} + \alpha_p L$, where F_s and L denote the friction force and the normal load on a representative surface element, respectively, whereas the systemdependent σ_0 and α_p stand for an offset and a proportionality coefficient [1]. This constitutive relation holds all the way down to the nanometer scale in the presence of lubricants or contaminants with values in the order of 0.1 leading to a reduced but not vanishing friction [34]. We will explore the possibility of contact area being important in a separate study.

As far as the wavelength dependency is concerned, we suppose that this experimental result may be traced back to the mean square slopes of the measured profiles. The mean square slope S_{rms} shows a maximum value for a structural wavelength of 5 µm whereas the smallest S_{rms} can be found for 18 µm. This value is in leading order inversely proportional to the real contact area [35]. On the other hand, the observed friction reduction may also be a consequence of fabrication errors during the patterning process. The resulting laser patterns are not perfectly sinusoidal and thus the interlocking could be complicated for a 0° alignment and a decreasing structural wavelength.

In general, the tribological behaviour of austenitic stainless steels is strongly affected by the formation of strain induced ε -martensite [36]. According to Huebner et al. the martensitic transformation might occur by shear deformation of the lattice [36]. During sliding, the dislocation density increases and as a consequence, so does the stacking fault density. These steels have relatively low stacking fault energy of about 18 mJ/cm² which is comparable to Ag [37]. As already discussed, the stacking fault energy has a significant impact on the mechanical behaviour [38]. Like ɛ-martensite, the formed stacking faults are of hexagonal symmetry and thus finally pile up to form ε -martensite [36]. Remarkable work in this field has also been published by Rigney and Hirth. Rigney reported about the occurrence of large plastic strain fields during sliding deformation resulting in a strong rise of dislocations which organise themselves in dislocation cell structures [39]. Typical values for dislocation densities after sliding contact of ductile metals are in the range of 10^{15} l/m² which is consistent with simulation results from Kuhlmann-Wilsdorf [39, 40]. X-ray diffraction could not prove the existence of martensite after laser irradiation. However, further experimental work is necessary to study the potential formation of martensite and the influence of dislocations and stacking faults on the friction behaviour of the used steel before and after laser patterning as well as after the friction experiments by high resolution techniques. In particular, TEM and XPS experiments are a current matter of subject for more detailed studies of the microstructural and depth-sensitive chemical situation.

Conclusions

In conclusion, we have found that the dry friction between two laser-structured solids depends not only on the wavelength of the structuring but also on the relative orientation between the patterns. Surface chemistry and mechanical properties, such as hardness and yield strengths, can differ for different wavelengths and might – in parts – be responsible for why different wavelengths produce different friction. However, considering their influence cannot explain the critical role of alignment: in each experiment did the friction for misaligned surfaces turn out smaller than for a perfectly oriented interface, given the same wavelengths in both experiments. One possibility for the origin of the dependence on alignment is that the contact area is alignment dependent. We explore this idea in more detail in a companion paper [41].

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Figures



Figure 1: Schematic drawing of the interference setup. The SEM image shows a typical interference line-pattern made on stainless steel.



Figure 2: Two-beam interference line-pattern in a 1.4301 stainless steel substrate with a periodicity of around 9 μ m measured by light microscopy.



Figure 3: Topography plots for a patterned steel substrate (black) and a 100 Cr 6 tribometer ball (red) measured by WLI.



Figure 4: Surface profile plots of laser-patterned steel surfaces measured by WLI. The periodicity was set to be 5, 9 and 18 μ m.



Figure 5: SEM/FIB image of the pristine steel surface. The image was recorded by means of ion channeling contrast.



Figure 6: SEM/FIB image of the laser-treated steel surface which was recorded by means of ion channeling contrast. The laser intensity maxima correspond to the topographic minima with the clearly visible grain structure.



Figure 7: FIB cross-section of a steel surface after laser-patterning highlighting the grain morphology in the positions of maximum and minimum laser intensity. The dark grey layer on top sample surface represents a Pt protection layer resulting from the FIB cross-section preparation and is not representing the oxide film.



Figure 8: X-ray diffractogram of the unpatterned and laser-treated steel sample by grazing incidence X-ray diffraction. The structural wavelength was 18 μ m in this case.



Figure 9: Measured hardness of the reference and laser-patterned specimen surfaces with corresponding 3-D surface plots recorded by a Hysitron tribo-indenter. Only measurement points for the laser intensity maxima positions (topographic minima) are displayed. The structural wavelength was 18 µm.



Figure 10: Raman spectra measured on different topographic zones identified in figure 6. The laser spot size was 5 μ m and the information depth was ~1 μ m measured from the samples surfaces. Differences between the spectra indicate that the laser pattering irradiation induced a different oxidation state of the surface. The structural wavelength was 18 μ m.



Figure 11: (a) and (b) display a 3-D view of the relative alignment and sliding direction of the patterned contacting surfaces indicated by the black arrows. Figures 11 (c) and (d) show the temporal evolution of μ for different alignments (0°/90°) and two periodicities (5 and 18 μ m) compared to the unpatterned reference surface.



Figure 12: Light microscopy image of wear tracks with a 0° orientation between substrate and ball (a) and 90° (b).



Figure 13: Coefficient of friction over sliding time for three distinct line-spacings at 0° orientation.



Figure 14: Comparison of a laser-induced surface profile before and after sliding at a 0° orientation measured by WLI in (a). Figure (b) shows a SEM image of the worn surface pattern under a tilt angle of 52°. The structural wavelength was 18 μ m.

Parameter	rms (nm)	$S_{rms}(\mu m/mm)$	H (nm)	R _{sk}	R _{ku}
Unpatterned Reference	34 ± 5	26.32 ± 4	94.45 ± 11	-1.5 ± 0.2	3.12 ± 0.8
5 µm	326 ± 12	626.44 ± 57	860 ± 49	0.84 ± 0.03	3.59 ± 0.26
9 µm	305 ± 6	484.89 ± 36	775 ± 11	0.52 ± 0.07	2.38 ± 0.19
18 µm	354 ± 34	218.05 ± 15	1164 ± 107	1.45 ± 0.13	5.30 ± 0.53

Table 1: Topographic parameters of laser-patterned samples in comparison with the unpatterned reference recorded by WLI.