Evaluating Surface Cleaning Techniques of Stone Tools Using Laser Scanning Confocal Microscopy

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Introduction

Stone tool use-wear analysis reconstructs how tools were used in the distant past. This is accomplished through the identification of microscopic traces of polish, striations, and fractures left on the tool's surface. Use-wear analysis is based on the hypothesis that different contact materials (for example, wood, wheat, or meat) and different motions (for example, cutting, scraping, or drilling) produce distinct microscopic traces on the used tool, including microfractures, polish, and striations. Understanding the function of tools gives archaeologists insight into the behaviors of past peoples, allowing for the reconstruction of ancient ways of life.

Traditional use-wear analysis uses light microscopy to visually identify wear traces [1, 2, 3]. However, this qualitative analysis can lead to interpretive conflicts between researchers. In recent years, new methods for measuring use-wear have been developed that quantify functional traces on stone tools [4–12]. Many of these methods quantify the surface roughness of the polished areas, measuring topographic features at small scales to understand variation in surface texture created by contact materials such as antler, meat, and wood.

One method that shows particular promise uses a laser scanning confocal microscope (LSCM) (in this case, the OLYMPUS LEXT OLS4000) to characterize surface texture. Outlined here are the results of measurements taken with the above instrument to understand the effects of various cleaning methods on the surface roughness of experimental stone tools used to cut wheat. Currently, there is little consensus among use-wear analysts on how to adequately clean stone tools prior to analysis. Standardization is integral to the development of use-wear analysis and for continued research into the quantification of wear traces because it allows for comparability of the results from different researchers.

In the research presented here, experimental stone tools were subjected to three levels of cleaning: (1) with alcohol, (2) alcohol followed by soap and water, and finally (3) chemical cleaning with potassium hydroxide (10%) followed by hydrochloric acid (10%). These three stages of cleaning represent commonly used techniques of sample preparation and are increasingly invasive. The results of this study contribute to the standardization of sample preparation and illustrate the application of LSCM for archaeological use-wear studies. This article describes measurements that indicate how different commonly used cleaning protocols affect surface roughness measurements.

Methods

Instrumentation. The use of LSCM is becoming increasingly popular in archaeological studies of stone tool use-wear quantification [4, 5, 10]. This method has the ability to

characterize surface texture by constructing 3D models of surface features (Figure 1). Quantitative measurements of surface texture, including surface roughness, may be extracted for distinguishing polishes from different contact materials. The measurements taken with the LEXT OLS4000 LSCM adhere to ISO25178-2 for the measurement of areal surface features. Objectives lenses may include $5\times$, $10\times$, $20\times$, $50\times$, and $100\times$. In LSCM, images are formed by collecting reflected light from discrete focal planes using a pinhole system. In-focus slices from different z-heights are processed together to create a 3D model of the object's surface.

Wheat harvesting. In August 2011, harvesting experiments were conducted in Southern France (organized and directed by Dr. Patricia Anderson, Centre National de la Recherche Scientifique). The experiments took place over five days, with three days dedicated to cutting the field, one day to threshing the wheat, and one day to winnowing the grain. The harvested field, located outside the town of St. Vallier de Thiey, was planted with einkorn wheat (Figure 2). This wheat is an early domesticated species, making it a good analog for ancient harvesting activities.

The experimental sickle used for harvesting was modeled after an archaeological piece from the Natufian occupation at Kebara Cave (Israel) (Figure 3). The sickle was armed with six chert geometric microlith stone tools (Negev chert) and was

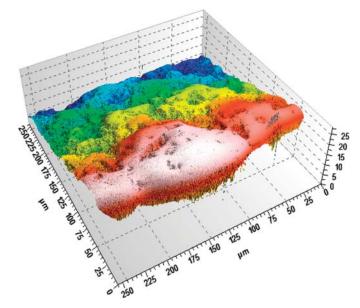
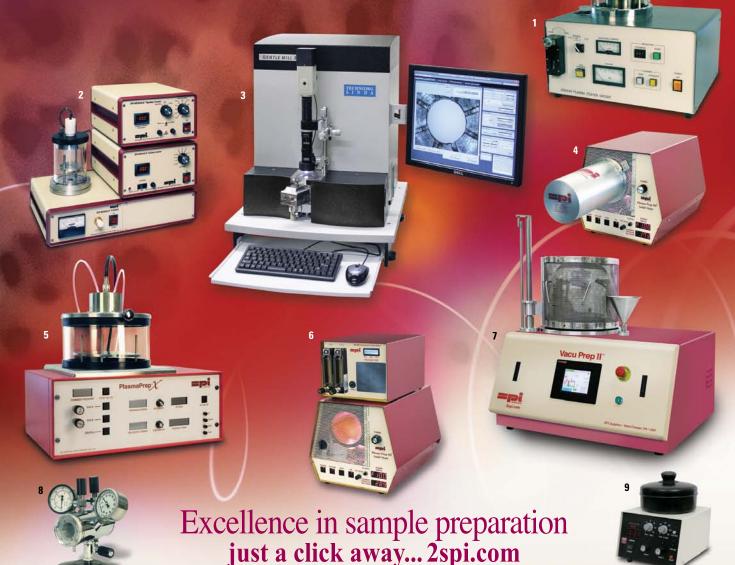


Figure 1: Three-dimensional image taken with a LSCM of a stone tool used for harvesting wheat. The tool's edge is oriented toward the bottom of the screen. Polish from wheat contact is represented by the white areas on the tool. Courtesy of J. Marreiros, J. Gibaja, and N. Bicho, Cambridge Scholars Publishing.

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used for approximately 12,000 strokes (approx. 400 minutes). Two microlith inserts from the sickle were chosen to test differences in cleaning protocols.

Measurement procedure. Two stone tool inserts from the sickle were chosen for analysis. In the first stage of analysis, the stone tools were cleaned with methyl alcohol and technical



Figure 2: The author harvesting wheat in a field located outside the town of St. Vallier de Thiey, France.



Figure 3: Experimental sickle used for the harvesting experiment. Roughness measurements were made on two of the chert inserts. Total length = 30.5 cm. Courtesy of J. Marreiros, J. Gibaja, and N. Bicho, Cambridge Scholars Publishing.

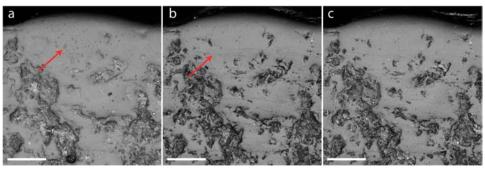


Figure 4: LSCM images of stone tool #1. (a) cleaned with alcohol, (b) cleaned with soap and water, (c) cleaned with KOH (10%) and HCL (10%). In image (a), a grease spot is indicated by a red arrow. The red arrow in image (b) highlights a fine striation produced by cutting motions. Scale = 40 μm.

wipes, minimizing the transfer of residue from the cloth to the artifact. Once the pieces were cleaned they were scanned with the confocal microscope using the $20\times$ objective and $50\times$ objectives, respectively (Figures 4a and 5a).

For the second stage, the stone tools were cleaned with warm water and a mild detergent (nutratek) while being lightly brushed with a soft-bristled toothbrush. After cleaning, they were again scanned in the same location and at the same magnification (Figures 4b and 5b).

During the final stage of the analysis, the stone tools were subjected to chemical cleaning (as outlined by [1]). First they were soaked in a bath of 10% potassium hydroxide (KOH) for 10 minutes to remove organic deposits. Following this cleaning, the lithics were soaked in 10% hydrochloric acid (HCL) for 10 minutes to remove any mineral deposits. Finally, they were bathed in clean water to remove any remaining chemical traces. Scans were taken of the microliths after the final cleaning stage (Figures 4c and 5c). Thus, the lithics were subjected to increasingly invasive cleaning techniques during each stage of analysis.

For each tool, five sampling areas of $30 \, \mu m \times 30 \, \mu m$ were selected for measurement from the polished surface of the lithic. The same five areas were scanned at each cleaning stage to ensure that the measurements reflected changes in the cleaning technique and not differences in surface topography. An average roughness measurement (Sa) was recorded for each of the five areas using MountainsMap* software produced by Digital Surf. The results of the five scans per tool at each cleaning stage were averaged to generate a mean surface roughness for each tool at each stage of cleaning.

Results

After alcohol cleaning. The overall results of the study indicate that alcohol cleaning is not sufficient for either visual interpretation of use-wear or for the quantification of polish surface texture (Figures 4a and 5a). Figure 6 shows that measurements of surface roughness for tool #1 cleaned with alcohol have a high variance. For tool #2, the piece had both a high variance and a high average roughness value (Figure 7). This is likely attributable to the presence of greases (which have a smooth texture) and particulate matter (which is rough) along with the texture found on the worn surface creating a highly variable surface roughness. This interpretation is further corroborated with the visual assessment of the images, where grease and other particles can be seen on the surface. These spots are visible on the highly polished areas of the tool and appear as gray spots on the surface (Figures 4a and 5a).

After soap and water. Cleaning the tool with a detergent removed the grease and much of the particulate residue, resulting in measurements with lower variance for both tools. Small striations resulting from the cutting motion are now visible on the surface, which were not visible when the tool was cleaned with only alcohol (Figure 4b). For tool #2, the mean roughness of the polished area for the tool cleaned with detergent was significantly lower than the mean roughness for alcohol cleaning, suggesting that

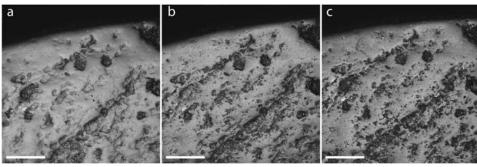


Figure 5: LSCM images of stone tool #2. (a) cleaned with alcohol, (b) cleaned with soap and water, (c) cleaned with KOH (10%) and HCL (10%). Scale = 100 µm.

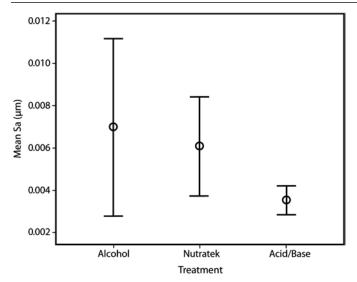


Figure 6: Average roughness (Sa) values for stone tool #1 after alcohol, nutratek, and chemical cleaning.

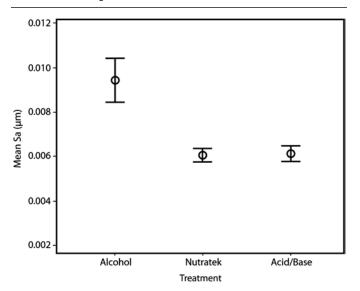


Figure 7: Average roughness (Sa) values for stone tool #2 after alcohol, nutratek, and chemical cleaning.

foreign material was removed from the surface, resulting in a less variable texture (Figure 7).

After chemical treatment. Finally, the surfaces cleaned with the acid and alkali method appear free from small particulate matter (Figures 4c and 5c). Thus, the texture

variation is reduced considerably in the surface roughness measurement of the chemically cleaned pieces. This is particularly evident in tool #1, where there is a clear reduction in the variance of the surface roughness between the surface cleaned with nutratek and the surface cleaned with chemicals. It is interesting to note that there is little difference in mean surface roughness between the cleaning with detergent and cleaning with chemicals for tool #2, suggesting that the soap cleaning was sufficient for this particular tool.

Discussion

It is important to develop standards for analysis as new methods for the quantification of stone tool use-wear analysis are developed. Currently, there is no consensus among researchers on how to adequately prepare and clean stone tool samples prior to analysis. The results presented here show that differences in cleaning practices do have an effect on measured surface texture. In addition, visual differences can be seen between the different cleaning stages, particularly for visible grease and particles on the tools cleaned with alcohol. This highlights sample preparation as an important area of methodological research for use-wear analysis.

The measurements for both tools cleaned with alcohol show the highest average roughness measurements and the most variance. This suggests that there is still a large amount of particulate matter adhering to the surface of the tools. The average roughness (Sa) measurements for tool #2 show that there is little difference between the average roughness of the tool cleaned with nutratek and the tool cleaned with chemicals. This indicates that the surface probably was not chemically altered by the acid/alkali cleaning procedure because the roughness values did not change between the two measurements. However, the measurements from tool #1 indicate that average roughness was lowered when the tool was chemically cleaned. Because the results from tool #2 indicate that the chemical cleaning did not alter the surface roughness, the authors suggest that the changes in measurements of tool #1 are from the removal of particulate matter adhering to the surface that was not removed with other cleaning methods.

Therefore, to ensure that surface texture is properly measured when doing quantitative use-wear analysis, we would recommend acid/alkali cleaning prior to imaging for metrological purposes. Clear differences in the measurements between detergent cleaning and chemical cleaning in tool #1 suggest that chemicals are needed to remove all adhering materials from the lithic surface. However, for visual assessment the tools cleaned with soap and water provided adequate cleaning if chemical facilities are not available.

Conclusion

This case study presents a new application of laser scanning confocal microscopy for the measurement of stone tool surfaces, contributing to the standardization of use-wear analysis. The cleaning techniques presented here can now be applied to archaeological assemblages to gain a better understanding of past behaviors through stone tool use.

Acknowledgements

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