

NANOTECHNOLOGY LEARNING MODULES AND ATOMIC FORCE MICROSCOPY
OF NEANDERTHAL STONE TOOLS

A Thesis
by
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ABSTRACT

NANOTECHNOLOGY LEARNING MODULES AND ATOMIC FORCE MICROSCOPY OF NEANDERTHAL STONE TOOLS. (May 2011)

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This thesis uses a Veeco Icon Atomic Force Microscope (AFM) to educate undergraduate students about the nanoscale world and to perform archaeological research. In chapter 2, an educational resource is developed to provide hands-on nanotechnology experience for undergraduate students. With the rapid growth of atomic force microscopy at many levels of industry and academia, it is important to expose the next generation to this technique. This learning module attempts to provide an experimental approach to learning about AFM phase imaging and its many applications. The module was field-tested by an upper-level undergraduate course (Experimental Methods in Physics) in the Physics and Astronomy department at Appalachian State University (ASU). Most of the students in the Experimental Methods class had never studied or worked with scanning probe microscopy previously, so their feedback helped to enlighten the developers about areas needing clarification.

In chapters 3 and 4, AFM is used as one of several techniques for classifying the use of Neanderthal flint tools from Weasel Cave, Russia. These stone tools were identified as being used for tasks such as wood working, hide scraping, and meat cutting. Depending on

the type of flint and the task involved, various degrees of abrasion occurred, leaving behind microwear polishes. These microwear traces are localized regions where the degree of polish is strongly influenced by the task being performed. In the past, most flint tool-use classification schemes were qualitative: a trusted expert performed a visual categorization using a stereo-light microscope. The research presented in this thesis attempts to advance the study of microwear analysis using both qualitative and quantitative techniques: incident light microscopy, AFM, scanning electron microscopy, and optical interferometry. Using statistical analysis of roughness, skewness, and kurtosis, measurable differences are shown between tools identified as being used for different tasks. This is exciting because it indicates the success of quantitative microwear analysis in determining flint tool use. This research is one of the critical steps to distinguishing microwear polishes using quantitative analysis techniques such as atomic force microscopy.

DEDICATION

This thesis is dedicated to my Father and Mother, William R Faulks and Ellen S Faulks. Thank you for giving me life, training me, loving me, and giving me an example to follow. Your lives have been my primary inspiration at every point. Every year, I become more thankful for you. Thank you for the sacrifice it took to love me selflessly for many years. The self-sacrificing love you have shown me has become a humbling picture of Christ's love for His church. Mom and Dad, you have blessed me in many ways, but none is more important than this: teaching me of Christ's life, death, and resurrection, and then backing up these claims with lives that have been changed by His Gospel.

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CHAPTER 1: INTRODUCTION AND LITERATURE REVIEW

BACKGROUND OF ATOMIC FORCE MICROSCOPY

In 1972, a group from the National Bureau of Standards developed the Topografiner, a microtopography instrument with resolution approaching the atomic scale (Young 1971; Young *et al.* 1972). This instrument used a sharp probe controlled by X, Y, and Z piezos to slowly scan a metal surface, with the strict condition that it not come in contact with the surface. These piezoelectric elements respond with a mechanical strain to an applied voltage, with step-sizes on the atomic scale. The height of the probe above the surface was held constant by maintaining a constant tip-sample current. A small quantum tunneling current would leak from the probe to the surface, and secondary electrons were detected with an electron multiplier. The Topografiner helped to close the gap between surface metrology and theoretical surface science.

In 1981, Binnig and Rohrer developed Scanning Tunneling Microscopy (STM), a technique capable of obtaining true atomic resolution images of metals (Binnig *et al.* 1982). For the first time ever, quantum vacuum tunneling was used for surface microscopy, achieving atomic scale topographic images. When a voltage is applied to an atomically sharp probe very near the sample surface, a tunneling current is created. In STM, a tunneling current that depends strongly on the distance between the two electrodes (tip and surface) is established. The tip is raster-scanned across the surface, while a feedback loop controls the piezo voltage, adjusting the tip-sample separation to maintain constant current. The piezo voltage is recorded. By calibrating the instrument with samples of known topography, the piezo voltage can be related to height, and a topography map of the sample can be generated

by the computer. The STM quickly gained acceptance and in 1986, Binnig, Rohrer, and Ruska received the Nobel Prize in physics.

Soon after, Binnig *et al.* published another ground-breaking study, introducing the atomic force microscope (Binnig *et al.* 1986). Initially, this instrument was labeled as a mere extension of STM, but it soon grew into a mainstream microscopy used in multiple disciplines with a variety of diverse applications. The first AFM employed an STM to monitor the deflection of an elastic cantilever as it was displaced by a very small load. An atomically sharp probe was placed near the end of the cantilever, functioning as the contact point for force measurement between the tip and the sample. Forces down to 10^{-18} N were predicted, corresponding to theoretical measurable distances as small as 10^{-4} Angstroms. This was a significant and startling prediction because interatomic forces range from 10^{-7} N for ionic bonds to 10^{-11} N for van der Waals forces. Thus, the theoretical sensitivity of the instrument indicated that all important interatomic forces should be measurable.

Cantilever fabrication and selection was an important technology to enable AFM. Springs constants need to be as small as possible for large deflection, but large enough to minimize vibrational noise. Cantilever resonant frequency can be approximately modeled as:

$$f_{res} = \left(\frac{1}{2\pi}\right) \sqrt{\frac{k}{m_o}} \quad (1)$$

where k is the spring constant and m_o is the effective mass of the spring load. Ultimately, spring constants were chosen to be on the order of several N/m, leading to resonant frequencies in the range of tens of kHz.

Soon after it was introduced, Binnig *et al.* achieved atomic resolution on insulating (boron nitride) and conducting (graphite) surfaces, demonstrating the flexibility of this technique (Binnig *et al.* 1987). Individual atoms were distinguishable with 2.5 angstrom

lateral resolution in the graphite sample, and similar resolutions were achieved in the boron nitride sample. The microscope was primarily limited by thermal fluctuations and asymmetry in the tip. The surface plot is a close representation of the actual surface, but the tip shape influences the accuracy of this measurement. The radius of curvature of the tip is a finite size (often on the order of 10 nm), limiting the precision of the replication of the actual surface morphology.

Initially, all AFM scans were performed using contact mode. Although many other techniques have evolved since then, contact mode remains one of the most popular scan modes. In contact mode, the tip is raster-scanned across the surface, being deflected as it encounters topographical features. The tip position is continuously adjusted to maintain a constant cantilever deflection. The height adjustment necessary to maintain this constant deflection is recorded as the data.

One challenge encountered with contact mode is the frictional force between the probe atoms and the surface atoms. In ambient scanning conditions, several tens of monolayers of gas and water vapor typically adsorb on the surface. When the tip comes in contact with this contamination layer, the cantilever is pulled toward the sample surface by surface tension. The magnitude of this attractive force varies, but is typically on the order of $10^{-7}N$. Although lateral force microscopy (LFM) uses these frictional forces as an imaging signal, the probe may cause possible damage to biological samples and decreased resolution at the nanoscale. One solution to these challenging problems emerged when microscopists introduced non-contact mode. The piezo is driven with an AC wave as the tip hovers in the attractive van der Waals region several nanometers above the surface. These attractive forces

cause damping as the cantilever is oscillated, and a surface map is generated. These damping forces cause changes in amplitude and phase, and either of these may be measured for data.

After several years, another popular imaging mode was developed for AFM. Tapping mode was patented by Digital Instruments and was demonstrated to yield high resolution images (Umemura *et al.* 1993; Zhong *et al.* 1993). The cantilever is driven at or near its resonant frequency (typically between 50 – 500 kHz), with an amplitude ranging from a few nanometers to several tens of nanometers. The tip is lowered until the oscillation is slightly damped by interactions with near-surface forces (van der Waals forces, capillary forces, magnetic forces). AFM tapping mode is normally operated with a constant amplitude feedback loop, and the piezo adjustment necessary to maintain this parameter is recorded as data (Cleveland *et al.* 1998). Tapping mode has several advantages over contact mode, including the reduction of frictional forces due to dragging the tip across the surface. This helps to reduce the wear of the tip when imaging rougher samples. When tapping mode was introduced, delicate polymer, silica, and cellular surfaces were successfully imaged without damaging them, allowing for the expansion of AFM into many new fields.

AFM detection systems and feedback loops must be capable of monitoring tip displacements of 1 angstrom or less to obtain true atomic resolution. The first AFM systems used an STM mounted above the cantilever. As the tip probed the surface in contact mode, the STM measured the changes in tunneling current and recorded this data. Atomic resolution was obtained with this method, but only small variability in the surface-terrain was permitted (a few nm) because of the exponential sensitivity of tunneling current to distance. Another drawback to the STM method of detection was the build-up of contaminants on the cantilever surface, reducing the tunneling current. Within several years, a laser-detection system was

developed and became the industry standard continuing to this day (Alexander *et al.* 1989; Butt *et al.* 1990; Meyer and Amer 1988; Rugar and Hansma 1990). The top of the cantilever was treated with a highly reflective coating, and a laser beam bounced off this reflective surface up to a 4-quadrant photodiode. The signal difference in each quadrant was measured and then correlated with the cantilever deflection using geometry. The cantilever is modeled as a Hookean spring for small displacements, so the tip-sample force can be calculated, given a knowledge of the spring constant.

In the two decades since AFM was invented, its applications have extended into many new fields. Due to its ability to image at the nanoscale and, in newer versions (i.e. Veeco Dimension Icon), also at the microscale, one instrument can be used for many applications. Most AFM manufacturers offer peripheral attachments, giving the user access to a whole slew of SPM capabilities. Some of the most popular techniques include, but are not limited to: STM, Scanning Capacitance Microscopy (SCM), Lateral Force Microscopy (LFM), Electrostatic Force Microscopy (EFM), Magnetic Force Microscopy (MFM), and Nanoindentation.

FORMER WORK DEVELOPING MICROSCOPY EDUCATIONAL RESOURCES

Since the evolution of SPM technology, the development of educational resources has been important to complement microscopy research. As SPM becomes more widespread in academia and industry, it is necessary to provide educational structures and materials in parallel to support these microscopy applications. Technologies of this sort not only affect the relatively small number of users, but also the large body of manufacturers, educators, suppliers, and consumers. The production and spread of educational resources is an essential benchmark for the further integration of SPM into the modern world.

Some groups have made microscopy educational efforts using remote operation to allow virtual hands-on control of a microscope. Using a standard high-speed internet connection, users are handed control of a limited set of microscope parameters. The Bugscope outreach project (Potter *et al.* 2001; Wallace *et al.* 2008) connects classrooms with an Environmental SEM (ESEM) where they may investigate an insect specimen of their choice. The lower vacuum of ESEM helps prevent charging in non-conducting samples such as bugs. The only drawback to the success of this project is the rather large logistical staff, indicating large amounts of required external funding.

A group from Arizona State University developed a remote-control SPM program for high school and undergraduate classes (Ong *et al.* 1999; Ramakrishna *et al.* 2000). The Interactive Nano-Visualization for Science and Engineering Education (IN-VSEE) project gives users access to real-time control of an SPM instrument. Also known as the SPM-LIVE! project, this well-established microscopy resource extends the capabilities of this high-tech laboratory to any classroom with an internet connection around the world. This outreach structure brings students as close as possible to the nano-scale world without actually stepping foot in a research laboratory.

Another group specializing in remote AFM operation has made use of a nanoManipulator peripheral device, allowing high school students to see and touch viruses *in vivo* (Jones *et al.* 2003; Jones *et al.* 2004; Jones *et al.* 2006). In addition to observing real-time manipulations of the virus, students could even “touch” the virus using a responsive joystick with feedback linked to the forces in the AFM. After participating in this program over the internet, students were more likely to understand the size scale associated with microscale interactions.

Interactive microscopy learning experiences endow the next generation with a fundamentally different way of viewing the world at the micro and nanoscale. Despite the resources available through the WWW environment, more hands-on approaches to microscopy and nanotechnology education are needed to bring excitement and realism to these new curricula (Lehmpuhl 2003; Turner *et al.* 2006; Uddin and Chowdhury 2001). This thesis attempts to develop and test a new hands-on AFM learning module for undergraduate students at Appalachian State University.

FORMER QUANTITATIVE LITHIC MICROWEAR STUDIES

The recognition of a Stone Age (or Paleolithic) as the first evolutionary stage by humans was made by the Englishman John Frere for stone handaxes at Hoxne, and the Dane Christian Jürgensen Thomsen for his “Three Age System” (Trigger 1989). It is now recognized that the invention of flaked stone tools was the second significant step in human evolution, following that of bipedalism (Klein 1999). In an effort to discover more details about this earliest phase of human evolution, archaeologists have long been challenged with interpreting the function of these stone tools. Many specimens have been discovered worldwide, but it was not until the 20th century that scientific methods developed enough to perform accurate, repeatable experiments investigating stone tool use (Schick and Toth 1993).

Microwear analysis is a powerful technique that consists in the observation of stone tool microwear traces with those on experimental tools using stereo- and incident-light microscopes. Microwear analysis began with the “traceological” approach of Semenov

(1964) and was first reproduced in the West by Lawrence Keeley (1977, 1980). Initially, most investigations began at the qualitative level, where an expert in the field compared the morphology of microwear traces on a tool having known use (usually experimentally produced) with the morphology of microwear traces on the tool in question. These pioneering microwear methods focused on the function of Paleolithic tools made of flint (SiO_2) – a subtype of chalcedony, which is a sedimentary variety of quartz (Prinz *et al.* 1978). These qualitative methods served microwear analysts well for decades, but soon after Keeley's comprehensive methods were developed, new quantitative characterizations were sought after.

Near the turn of the 21st century, several microscopy and interferometry techniques were explored for their use in quantitative microwear analysis. Atomic force microscopy was introduced because of its high resolution and ability to obtain mean surface roughness for microwear polishes (Kimball *et al.* 1995, 1998; Schlichting 1997). These first studies demonstrated that many polishes have a surface roughness that is quantitatively distinct from the unused control region on the flint tool. Through this project, AFM was shown to be a promising technique for quantitative lithic microwear. Soon after, optical interferometry was used to characterize and differentiate polishes formed by working different materials (Anderson *et al.* 1998; Gonzalez-Urquijo and Ibáñez-Estévez 2003). When variables were controlled, this method helped to increase the objectivity of the microwear analysis.

In 2001, a series of publications was initialized, investigating lithic use-wear with a laser profilometer (Stemp *et al.* 2001, 2003, 2010). This variable-length scale technique measured surface roughness parameters and interpreted the roughness using fractal dimension. Results from laser profilometry showed distinguishable differences between several samples with

unique wear histories. Another study of notable significance was performed using laser scanning confocal microscopy for microwear analysis (Evans and Donahue 2008). Despite the similarities to SEM and light microscopy, this technique harvests true quantitative surface roughness data. Laser scanning confocal microscopy is an excellent use-wear analysis tool, yielding repeatable quantitative results, and demonstrating distinctions between different wear polishes.

DIRECTION OF THIS RESEARCH

This thesis is concerned primarily with two areas: development of an AFM learning module, and using AFM as a quantitative lithic microanalysis technique. The AFM learning module is developed in chapter 2, and it focuses on AFM phase-contrast imaging as a tool for topography and compositional differentiation. This chapter follows a format in which background research is presented, the motivation for the module is explained, the learning module is presented in full-text, and results are analyzed. Chapter 3 focuses on using AFM as a viable method for quantitatively distinguishing microwear traces on Mousterian flint tools from Weasel Cave, Russia. Chapter 3 was submitted to the peer-reviewed journal *Scanning* for publication in February 2011. In chapter 4, morphology of microwear traces is analyzed qualitatively (incident-light microscopy, scanning electron microscopy) and quantitatively (optical interferometry and atomic force microscopy) to ascertain a holistic understanding of polish development. Chapter 4 was presented as a paper at the Society for American Archaeology (SAA) conference in March of 2011. As stand-alone publications, both chapters 3 and 4 follow a format in which former studies are introduced, painting a

picture of the current state of lithic microwear analysis. Then experimental techniques are identified, results and analysis are presented, and conclusions leading to future work are discussed. As a whole, this thesis aims to expand the knowledge and application of AFM, furthering several fields of science.

CHAPTER 2:

DEVELOPMENT OF NANOTECHNOLOGY LEARNING MODULES USING AFM

BACKGROUND

RP Feynman initiated an ambitious effort to create functional technology at the nanoscale (Feynman 1959). Beginning almost immediately, the discipline of nanoscience sprang to life creating new technologies that continue to push the barriers of knowledge today. The term nanotechnology was first used in 1974 by N Taniguchi to describe the production and control of materials at the atomic or molecular scale (Taniguchi 1974). In the decades since this term was coined, the concept of atomic scale manipulation has remained the same, but out of necessity this is an interdisciplinary effort, expanding to many disciplines besides physics.

It is widely acknowledged that nanotechnology is spreading throughout society (Dang *et al.* 2010; Paull *et al.* 2003; Schulte 2005). Most people in developed nations hold some opinion or ethical assumption about its goals and implications for life on earth. Opinions range from extremist neo-Luddism to enthusiastic technophilism. Academia has researched and published on nanoscience at an increasing rate during recent decades. From 1989 to 1998, the number of nanotechnology publications increased from 1,000 per year up to more than 12,000 per year (Hullmann and Meyer 2003). Large companies have made huge investments for the sole purpose of manufacturing materials at the nanoscale (Draper Fisher Jurvetson, Harris & Harris Group, Nanotech Partners, NGEN Partners). Many technologies in the military, medicine, and even in the home rely on manufactured interactions that are only possible at the nanoscale. All signs point to a developed world in the near future that not

only accepts nanotechnology as a normal part of life, but even depends on nanotechnology to make significant advances in areas of health, electronics, manufacturing, and many other scientific fields.

In 2001, President Clinton signed into existence the National Nanotechnology Initiative (NNI), a collaborative effort to stimulate and coordinate nanotechnology R&D in the U.S. According to a recent publication, the vision of NNI is “a future in which the ability to understand and control matter at the nanoscale leads to a revolution in technology and industry that benefits society” (NNI Strategic Plan 2011). Since its inauguration, the NNI has influenced or backed much of the research in this thriving field.

In 2004, the NNI established four goals to track the success of its vision. One of these major goals is to “Develop and sustain educational resources, a skilled workforce, and the supporting infrastructure and tools to advance nanotechnology.” This goal is different from the other goals: it concerns the development of people, the next generation of researchers, while the other goals concern the development and implementation of the technology itself. This is necessary to truly achieve any more significant breakthroughs in the development of nanotechnology. This NNI goal highlights a critical need: nanotechnology is fundamentally different from other technology, and the educational programs needed to equip students will be fundamentally different from traditional science education. Nanotechnology uses material properties and manipulation at the < 100 nm scale, and a new pedagogy is necessary to teach about these effects.

To prepare the world adequately for the certain advancement in nanotechnology and nanoscience research, the education system must equip K-12 students with the frame of mind

necessary to comfortably explore and utilize material properties available at the nanoscale. The need for nanoscience education is two-fold in that it will prepare students for the nanotechnology they will face in the world, and it will fill the manufacturing and service jobs that nanotechnology continues to create (Committee for the Review of the National Nanotechnology Initiative 2002). Students must be progressively prepared for STEM careers (science, technology, engineering, and mathematics) which are becoming increasingly necessary and competitive. The effects of nanotechnology are not limited to the academic community alone. Even students who pursue non-STEM careers will be affected by the progress made within nanotechnology during coming decades. From chemical and biological nanosensors to nanoelectronics within computing, students today will encounter technology relying on nanoscale properties. Allowing students to do hands-on experiments and measurements is a critical next-step to improving nanoscience education. It is difficult for most people to understand atomic and molecule-scale interactions because they are limited by their imagination in most cases. Hands-on nanotechnology education resources give students a tremendous advantage, because they can move beyond imagination to investigation. Students are more excited about learning when they can see and touch the technologies they hear about in the classroom.

One group from the University of Washington recently recognized the need for hands-on nanotechnology training. The Nanotechnology Undergraduate Education: Using Nanoscience Instrumentation for Quality Undergraduate Education (NUE UNIQUE) provides sophomore and junior undergraduates with a lab experience in scanning probe microscopy (SPM) (Overney and Sarikaya 2009). The NUE UNIQUE curriculum provides mobile workshops and lectures in topics such as AFM, Dip-Pen Nanolithography (DPN),

Scanning Tunneling Microscopy (STM) and several other imaging modes. Students are challenged to apply many of the theories from former lecture courses in this one-week educational journey into the world of SPM nanotechnology.

Groups from universities around the country have successfully integrated SPM educational courses into their standard curriculum (Adams *et al.* 2004; Glaunsinger *et al.* 1997; Sullivan *et al.* 2008; Zhong *et al.* 2003). Some groups have even bridged the gap between institutions desiring hands-on nanotechnology education, yet lacking the resources to purchase an SPM system (Jones *et al.* 2004; Ong *et al.* 1999; Potter *et al.* 2001; Ramakrishna *et al.* 2000; Wallace *et al.* 2008). Remote SPM operation through the internet is more available than ever before, giving almost any K-16 school the access to the newest microscopy instrumentation. Remote learning is a useful tool, but true hands-on learning remains the most effective means of microscopy education. Much work remains to be done, to adequately prepare the next generation of scientists to understand and continue nanoscience research.

MOTIVATION

In the Fall of 2008, the Physics and Astronomy department at Appalachian State University (ASU) acquired two atomic force microscope (AFM) systems for use in teaching and research labs. These microscopes were purchased with funds from a National Science Foundation award (DMR 0821124). The Nanosurf Easyscan 2 is a mobile AFM system that can be transported to other classrooms where students can interact with the samples and can control the microscope for themselves. This type of outreach event has proven to excite

students at local schools in grades K-12 about nanotechnology and the tools used to observe it. The Veeco Dimension Icon is a research AFM with multiple scan modes including contact mode, tapping mode, nanoindentation, and Harmonix mode. This AFM has already been used widely by faculty, students, and commercial users for nanoscience investigation. AFM is one of the most popular methods used to study nano and microscale surfaces. During the last two decades, AFM has come alongside older, established methods such as scanning electron microscopy (SEM) and scanning tunneling microscopy (STM) to provide true topographical information about many surfaces.

One of the primary purposes for purchasing these AFM systems was to educate students about nanotechnology. Most people never have the opportunity to understand the nanoworld, let alone using state-of-the-art equipment for exploration. When students are introduced to nanotechnology through hands-on investigation, they are more likely to be excited about learning than when a pure classroom approach is the only method. From summer 2009 to present, these microscopes have been used in dozens of outreach projects for groups ranging from elementary school up through undergraduates. In addition, the research group developed and field tested three new AFM learning modules entitled, “What Makes a Diffraction Grating Work?” (Coffey *et al.* 2010), “What is Smooth?”, and “Magnification.” These three new learning modules introduce K-16 students to an AFM and expose them to general concepts in microscopy, such as magnification. Using these hands-on modules, students discover a new world, the nanoscale world. Students learn how to use an AFM, and they see that it can be used to scan and image objects at a very small scale. The learning modules also help to show students how macroscale phenomena (such as diffraction or how rough an object feels to the touch) can be explained by microscale and nanoscale

morphology. These three learning modules have been tested extensively and have proven effective at teaching and exciting students about nanotechnology.

These three original modules were successful when presented to K-12 students in classrooms and outreach events. However, students at the undergraduate level were not adequately challenged. Several classes in the undergraduate physics curriculum are ideal for implementing nanoscience experiments. Experimental Methods in Physics is a core class required for physics majors to graduate at ASU. In this class, students must plan, perform, and present both classical and modern physics experiments. We set out to develop a nanoscience experiment to serve as a platform for undergraduates students to begin using the AFM. This experiment would be integrated into the Experimental Methods course, giving undergraduates hands-on experience with state-of-the-art nanotechnology.

One of the challenges in developing learning modules for an advanced undergraduate lab is the steep learning curve inherent with AFM and most other types of microscopy. One of the goals of the Experimental Methods class is for students to gain independence in carrying out a scientific experiment. However, students cannot simply be unleashed with a sensitive microscopic instrument such as AFM. Lab groups are given 3 weeks to complete each experiment, which is not enough time to become competent and independent in a technique such as AFM. The risk involved in giving students total independence with the AFM must be weighed with the potential educational benefits of leaving them to perform the experiment on their own. We wanted to maximize students' exploration of the AFM hardware, software, and techniques, while giving them an appropriate level of guidance through the learning module text and personal interaction.

As observed in many undergraduate physics lab/lecture courses, students understand concepts better and are more excited about learning when they can connect a mental picture with an interactive learning experience (Uddin and Chowdhury 2001). When students take ownership of a physics lecture concept by applying it in lab to solve a real problem, they are more excited and engaged in the learning process. We applied this theory to the development of learning modules. Most of the mechanics and feedback loops in AFM are taught in other undergraduate physics courses. Students can apply their mathematical knowledge of simple harmonic motion, resonance, damping, and van der Waals forces to understand AFM operation. Students can understand complex tip-sample interactions by applying well-known classical and modern physics solutions.

In the process of developing the learning module, several experiments were considered for the purpose of introducing students to the AFM. This learning module was built around AFM phase-imaging because it is relevant to microscale and nanoscale science. The phase-imaging data collection mode goes beyond simple topographical features to reveal information about surface composition. Students are challenged to understand AFM operation, to obtain phase-images, and to interpret these images.

The AFM phase-imaging module is shown below in its full text. This learning module follows a format where the topic of study is introduced, background information is discussed, the activity is presented, and analysis questions are posed. An average lab group of 3 students typically completes the AFM phase-imaging module in 2-3 three-hour blocks of time. Students in the Experimental Methods class then present their work in the form of a research paper, poster, or other visual presentation.

LEARNING MODULE TEXT

I. Purpose

To understand how a phase image is acquired in an atomic force microscope (AFM), how to properly analyze a phase image, and what factors contribute to phase surface mapping.

II. Materials

- Veeco Icon (Atomic Force Microscope)
- Several pens, markers, highlighters, etc.
- A smooth sample surface to mark on (such a plastic CD jewel case)

III. How does the AFM work?

An atomic force microscope uses a combination of a tiny cantilever, laser and photodiode to image a region at the micro or nanoscale. As the tip scans across the surface, the laser beam reflects from the back of the cantilever up to the 4-quadrant photodiode (Fig. 1). The information from the photodiode is then sent to the control electronics. Angular displacement of the cantilever causes one quadrant of the photodiode to collect more laser sum signal than the other quadrants. The feedback loop in the control electronics adjusts the vertical position of the tip to try to maintain the laser at the center of the photodiode. By knowing the distance the cantilever must be adjusted to maintain the constant photodiode output, the software can produce a topographical image of the surface as it scans point-by-point across the sample.

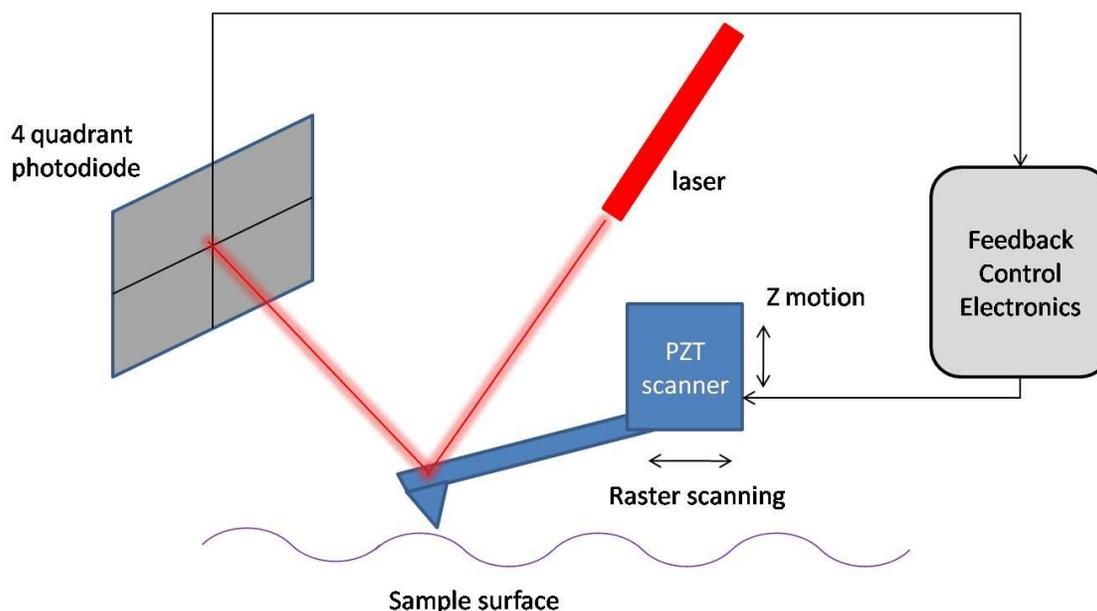


Fig 1. AFM schematic showing the laser, cantilever, and photodiode cooperating within the control feedback loop.

The atomic force microscope can be operated in many different modes. The most common ones are contact mode and tapping mode. Tapping mode will be used in this experiment. When an AFM is set in tapping mode, the cantilever oscillates very quickly up and down (the resonance frequencies of most cantilevers range from 10-200 kHz). The tip on the cantilever literally “taps” the surface gently. The feedback loop will adjust the position of the tip to maintain a constant amplitude of oscillation. Review the provided supplementary materials for a more in-depth explanation of AFM (Nanoscience Instruments, Inc 2011).

IV. What is the phase image?

The AFM typically acquires several types of data simultaneously as it scans the sample – height, amplitude error, and phase images. Each image highlights different features on the

surface. The height image provides a true topographical map, the amplitude error highlights the sharp edges, and the phase image indicates when a surface property changes.

The phase image goes beyond simple topographical mapping to reveal composition, adhesion, friction, viscoelasticity, and perhaps other properties. Applications include identification of contaminants, mapping of different components in composite materials, and differentiating regions of high and low surface adhesion or hardness (Li 1997). When the AFM is operating in tapping mode, it acquires the phase image by measuring both the input drive frequency from the piezo, and the resultant cantilever oscillation frequency. The phase image is generated by computing the phase lag from these two frequencies as seen in Figure 2. Phase lag typically occurs when tip-sample interactions such as electrostatics and surface viscosity cause damping relative to the piezo drive frequency (dashed wave). These signals are measured simultaneously by the NanoScope feedback controller (Bruker AXS 2011).

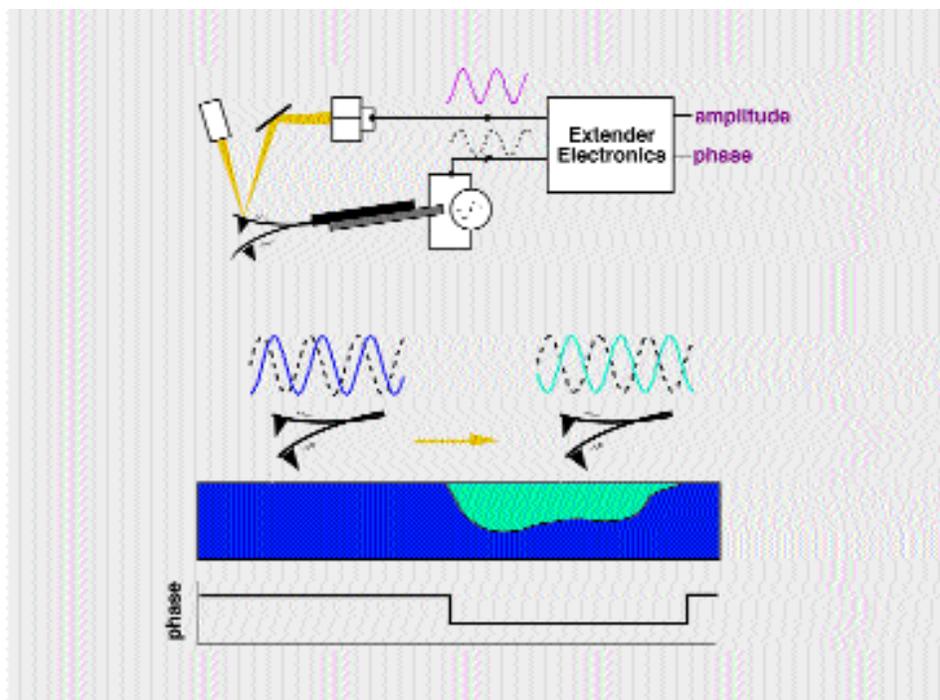


Fig 2. Phase imaging measures the phase lag of the cantilever oscillation (solid wave).

V. Why is the phase image useful?

The height and amplitude error images provide information about topography and sharp edges, but provide very little indication of surface composition. The phase image, however, is very sensitive to surface inhomogeneities and contamination (Stark *et al.* 1999). Thus, in a heterogeneous sample, the phase image will distinguish between the various materials.

According to the AFM resource library online, “Phase images often compliment topography images by mapping the various regions of the sample surface, each of which interact with the tip in a slightly (or significantly) different way from each. This difference is sometimes so subtle that it is barely noticeable in the topography image, but clearly visible in the contrast variations in the phase image. More often than not, however, topographic features convolve into the phase image, and must be recognized apart from the contrast in the phase image that

is primarily a result of material inhomogeneity” (Agilent Technologies 2011). Essentially, this excerpt says that although the phase image is very useful for discerning various regions on a sample, it does not provide true topographical data.

It is important to note that although the phase image *does* indicate material differences, it does not indicate what *type* of differences may be present. There is no simple correlation between phase contrast and a single material property (Bruker AXS 2011). Phase imaging is not well-understood, and it is difficult to quantify a specific phase lag for each material property. However, some research has modeled the tip-sample interaction in the phase image while controlling various parameters (Winkler *et al.* 1996). They concluded that the phase shift is non-linearly dependent on the sample stiffness and the sample damping. Stiffer samples with larger Hookian spring constants cause smaller phase shifts. Similarly, samples with very little surface damping produce images with small phase shifts. As one would expect, higher phase shifts indicate larger amounts of energy dissipated between the tip and the sample (Cleveland *et al.* 1998; Tamayo and Garcia 1998). Typical phase shifts between drive force and cantilever oscillation are in the $\sim 90^0$ range (Burnham *et al.* 1997). However, obtaining an accurate analytical model of the phase image requires a very detailed knowledge of the tip-sample interaction.

VI. What do phase images look like?

The images below contrast the height image on the left with the phase image on the right, demonstrating some expected results from this experiment.

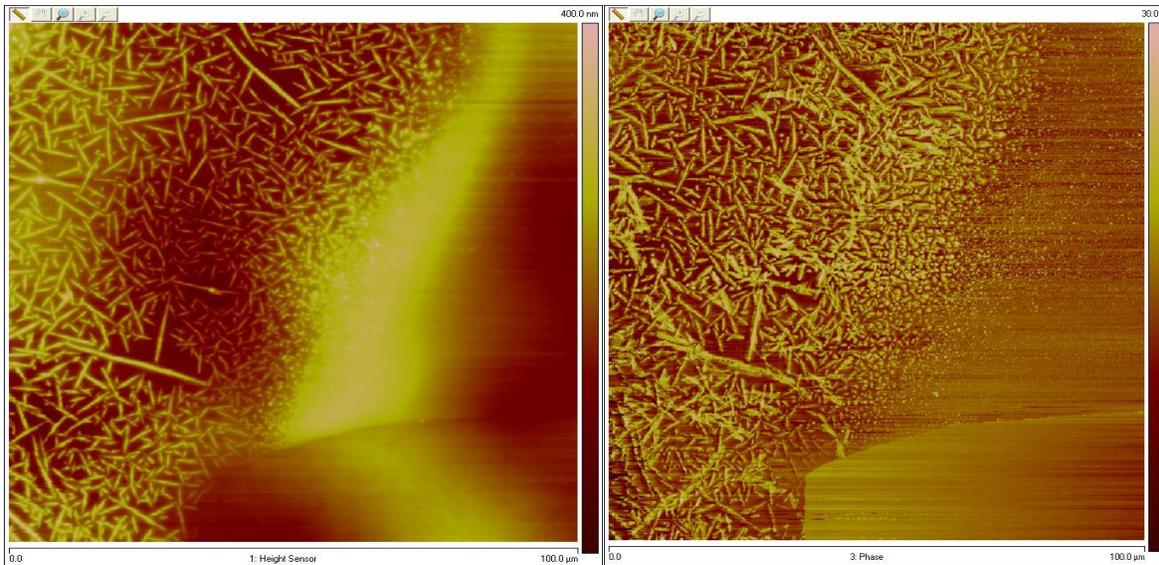


Fig 3. Height (left) and phase (right) images showing two ink marks on smooth plastic. The fibrous ink mark on the left side of each image is a purple marker. The smoother ink mark in the upper right is a red wet-erase marker. The region in the lower right is the plastic slide.

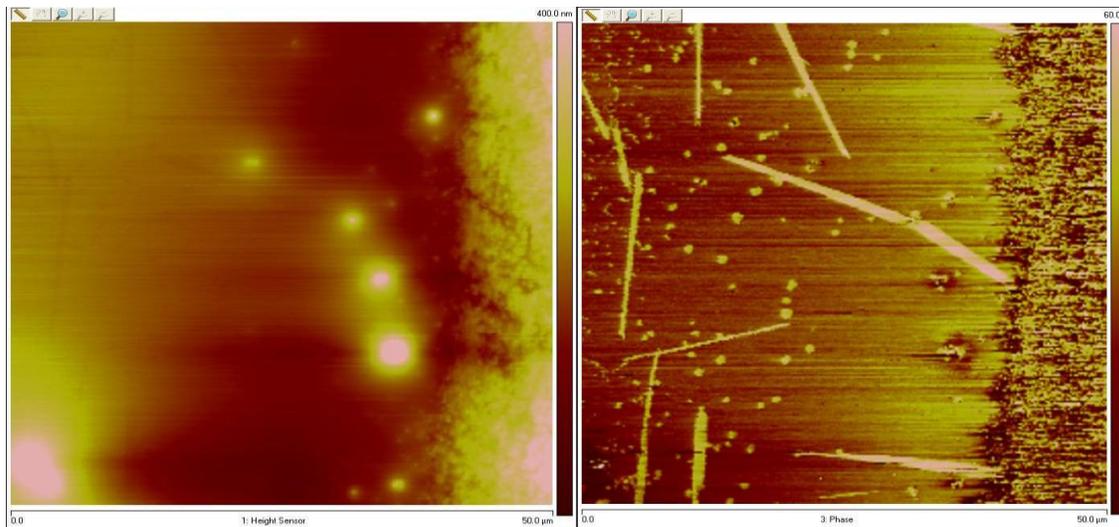


Fig 4. Height (left) and phase (right) images show a black pen mark covering most of the left side of each image, and the plastic on the far right side. The phase image indicates distinct inhomogeneities while the height image shows no such material differences.

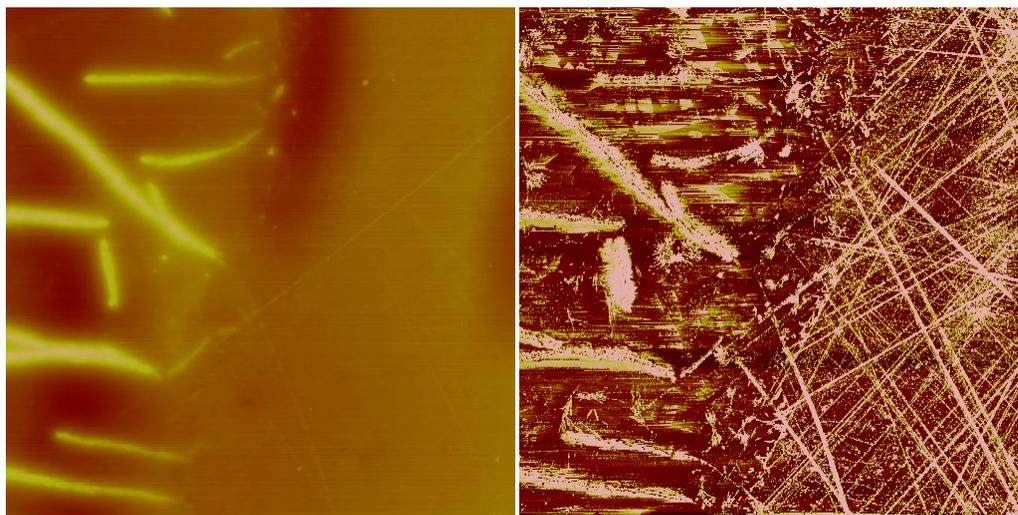


Fig 5. Height (left) and phase (right) images demonstrate how a phase image can glean topographical information that convolves in the height image.

VII. Procedure

- You will acquire images of samples with multiple regions of varying surface properties. This will maximize the contrast in the phase lag image. Ink from a pen or marker is a simple dye or pigment (often made from a carbon derivative) that can be used to observe an interesting phase image.
- During the microscope initialization, capture an image of the drive frequency sweep. Make sure to note the drive frequency set-point. Where is it in relation to the peak drive amplitude? Why was this drive frequency set-point chosen?
- Make a small mark on a piece of smooth plastic (such as a CD jewel case) and obtain a 100 μm x 100 μm image half-on, half-off the mark. The image should show the transition from the mark to the CD case. Try to image an area of the mark with a distinct, sharp transition between the two materials. Make sure to save the height, amplitude error, and phase images.
- Use the AFM software to make a topographical 1 dimensional line-plot across the phase image. It should pass through regions of varying phase lag.
- When presenting your data, include scale bars (horizontal and Z-scale) on all images. Show height and phase images together (see figures above).

VIII. Questions

- Do you notice a significant contrast between the various domains in the phase image?
- Based on the Z-scale, make a quantitative estimate of the typical phase lag for each domain. How much uncertainty is associated with your estimate?
- Does the relative phase lag in each region of interest provide any indication of surface properties? Explain.

- If possible, formulate a hypothesis as to the nature of the surface properties causing the phase lag in each domain. (Note: *Individual* surface properties typically convolve in the phase image, making it nearly impossible to discern quantitative information about the surface composition. However, the phase image can be used to form an educated guess concerning the contrast between materials.)

RESULTS

The AFM phase-imaging module proved effective at guiding ASU physics students in hands-on nanotechnology education. Twelve students used the learning module during the fall 2010 semester, and each of them provided feedback after completing the experiment. Several themes dominated student feedback about the AFM and the learning module. Most students responded with excitement about using a state-of-the-art AFM. They recognized the privilege of using this technology in an undergraduate class, and they were pleased with the results of the experiment. Most students also indicated that they were challenged to understand the AFM operation and data acquisition. With such a short amount of time given to learn the theories, techniques, and applications of AFM, students quickly realized they were only being briefly introduced to scanning probe microscopy.

Students appreciated the section in the learning module about phase-lag acquisition in AFM, but most students still felt overwhelmed when it came to thoughtfully analyzing the phase-images of the CD jewel case and the ink. Students seemed to understand the AFM phase-imaging process, but they had difficulty interpreting the phase-data. Specifically, most students were able to indicate some of the variables that could theoretically affect a phase-image, but could not synthesize these theories with their own data to form a supported

conclusion. For instance, some students noticed that when the ink was applied unevenly to the CD jewel case, these topographical inconsistencies caused some variation in the phase-image. However, most students could not convincingly describe in their final lab report how this topography change might cause a change in the phase-data.

Another trend in student feedback concerned the learning module's explanation of the AFM parameter controls. The Veeco Dimension Icon is a research-grade AFM, allowing for control of many parameters that students were not familiar with. For instance, many students did not understand the functional difference between P-gain and I-gain, causing them to feel like they were "fumbling in the dark" in one student's words. Other students felt that they did not receive clear guidance on controlling the amplitude set point and the drive amplitude. Several other control parameters caused confusion as well, and students expressed many doubts about the validity of their data, due to their inexperience with AFM operation. In actuality, most of the data was perfectly valid for their qualitative and semi-quantitative purposes, despite the concerns expressed by multiple students. However, it does appear that for advanced undergraduate students using an AFM, they should receive more explanation and training on the successful control of AFM parameters. Students should be given initial suggested values for operating the control parameters, and should then be expected to iteratively adjust the controls for their specific needs. Experienced AFM users sometimes describe successful imaging as an art, requiring creativity and adaptability to come alongside a knowledge of the theory of AFM operation. The ability to adapt to operating conditions occurs on a time-scale much longer than the few short sessions in which students used the microscope. Thus, students should be made aware that they ought to concern

themselves with understanding the basics of AFM operation, rather than honing their skills in AFM parameter control.

Some specific changes that should be made to the learning module in the future include an additional section introducing AFM control parameters such as gain, amplitude set point, drive amplitude, and scan speed. The learning curve is initially steep for many of these parameters, and students would benefit to read a few examples of how these variables can affect image acquisition. A more complete treatment of these parameters would also reduce student frustration because they would feel that they are applying their knowledge to understand AFM operation, rather than merely guessing. A further benefit of this additional section is the increased level of confidence that the students would have in their data.

CONCLUSION

The results of this AFM learning module indicate a successful landing within the architecture of the Appalachian State University (ASU) SPM education program. However, there are some next-steps to insure the continued growth of hands-on nanotechnology education at ASU. The theme of these critical next-steps is increasing the level of hands-on interactions that students have with microscopes available at ASU, such as AFM, scanning tunneling microscopy (STM), scanning electron microscopy (SEM) and focused ion beam (FIB). Some possible routes to implementation of this idea include, 1) Training more undergraduate students to do research on AFM and other microscopes, 2) Implementing more lab time in nanoscience courses offered here, and 3) Developing outreach programs, and even using undergraduate students to help lead these programs. All of these ideas for

future research are focused on decreasing the idle time of the microscopes and making them available to more students.

The process of developing nanotechnology educational resources is ongoing, due to the growing need for well-trained students in the job field (Roco 2004). New job opportunities are becoming available, and educational resources must adapt to equip students with specific skills and a broad knowledge of science (Uddin and Chowdhury 2001). As predicted by many trends surveying scientific, commercial, and government industries, creative implementations of nanotechnology continue to increase with each decade (Schulte 2005). Nanoscience improves people's lives around the world, and its multi-faceted benefits provide abundant evidence for emphasizing the development of learning resources.

Another well-established motivation behind the push for more nanotechnology education is the steady decay in U.S. science, technology, engineering, and math (STEM) education. Research over past decades (Hanushek and Kimko 2000) shows strong correlation between K-12 science and math test scores and a nation's long-term economic success. It is essential that governments, schools, and organizations place a unique emphasis on the development of STEM educational resources. If these alarming trends are to be reversed, it is necessary to provide innovative STEM education to students of all ages. Nanotechnology education provides students with functional skills, and it ought to become one of the main avenues for innovative STEM education to flow.

Popular opinions of nanoscience vary widely, and lack of public education is one of the factors slowing the growth of this resource. Without accurate and accessible sources, public opinions can plummet with a quick glance at an intimidating graphic or unformed

popular article. One of the markers on the road to implementing safe, effective nanotechnology is expansion of the education sector in schools and universities. Providing easily accessible public education is part of the framework necessary to further nanotechnology development.

CHAPTER 3:

ATOMIC FORCE MICROSCOPY OF MICROWEAR TRACES ON MOUSTERIAN TOOLS FROM MYSHTULAGTY LAGAT (WEASEL CAVE), RUSSIA¹

INTRODUCTION

Since flaked lithic technology was adopted 2.5-2.6 mya (Semaw 2000), stone tools served as a major adaptive means in early hominin evolution. Questions about how these stone tools functioned has been a major aspect of determining their role in human evolution. The first experimentally-based approach to the functional study of stone tool function was developed by Russian archaeologist Sergei A. Semenov (1957; translated into English in 1964) using both stereo- and incident-light microscopy. He was able to observe use-wear traces which, when compared with microwear traces on experimentally used replicas, permitted the identification of the kind of material being worked and the kinematics (or specific tool holding or hafting positions and the specific motions) of the tool. Through the relation of observed microwear traces on archaeological specimens to the same on experimental tools, the actual function of individual tools. This was largely possible because Semenov recognized that use-wear polishes varied according to the type of material worked (i.e., wood, bone, antler, ivory, hide, soft plant, etc.). However, these microwear polishes were only observable using the incident-light microscope.

The first replication of Semenov's observations in the West was by the American Lawrence H. Keeley (1977, 1980) using the incident-light microscope with magnifications of 50x-400x. The "Keeley Method" permitted the observation of additional microwear traces

¹ This chapter contains a paper submitted to *Scanning*, co-authored by Faulks, Kimball, Coffey, and Hidjrati.

and a more refined characterization of the polishes themselves. According to Keeley (1980) a microwear polish “can be described in terms of its brightness or dullness (that is, how much light it reflects) and its roughness or smoothness, as well as the presence of certain topographical features, like pits, undulations, and so forth.” Thus, reflectivity, roughness, and microtopography are the major axes of variation expected for microwear polishes. For example, Keeley (1980) described polishes in qualitative terms: wood polish is very bright, very smooth in texture with a gently domed microtopography. Bone polish is bright with a micro-pitted texture. Antler polish (Keeley 1980) is very bright and smooth, but with gentle undulations (“melted snow” appearance) when well-developed. Polishes resulting from the working of soft plants are very bright, spreading, with fluid distribution along the microtopography and exhibits “comet-shaped pits” and “filled-in striations.”

Polish resulting from cutting through meat is relatively brighter than the unaltered flint surface, its microtopography is clearly altered – “this luster seems to be the result of a smoothing of the microtopography on a very small scale. The normally grainy texture of a raw flint surface is replaced by a slight matt texture, which seems to preserve the very minute elevation and depression of the raw surface ... (Keeley 1980). Hide-working polishes were observed to vary according to moisture and fat levels: fresh versus dry hide. In both cases, the entire microtopography is affected; but fresh hide polish appears “greasy” somewhat like meat polish, while dry hide polish exhibits a rough/matt/dull texture which increasing with work (Keeley 1980).

At the same time, Keeley (1977, 1980) was able to show quantitative differences in reflectivity of these polishes in two clusters (soft plant, antler versus wood, dry hide, and

fresh hide) using a photometer and contrasting polish reflectivity for dark-field and light-field reflection in microamperes.

The revision of the Keeley Method used herein is referred to the Keeley-Plisson Method for it combines the experimentally-based system described in Keeley (1977, 1980) and systematically defined by Plisson (1985). Discrete microwear polishes are specifically defined for the attributes of polish *localization, extent, texture, contour, brightness, and coalescence/polish*. Kimball (1989) extended the Keeley-Plisson program and added hafting polishes as well, and a systematic, experimentally-based study of hafting traces is now published by Rotts (2010).

Today, the “high-power Keeley Method” is the most generally accepted method for the determination of stone tool function (Juel Jensen 1988; Yerkes and Kardulias 1993). At the same time, analyses of edge damage (Tringham *et al.* 1974) and residues (Fullagar 1998; Hardy 1999) are concurrent approaches, which are increasingly used in tandem with high-power analysis (Longo and Skakun 2008; Van Gijn 2010; Rots 2010).

In an unique collaborative study, Allen (Physics) and Kimball (Anthropology) demonstrated that the atomic force microscope (AFM) showed great promise in the analytical study of the function of stone tools. It was clarified by Kimball *et al.* (1995, 1998) (1) how microwear polishes formed; (2) how they vary quantitatively; and (3) how they can be visually characterized via new attributes. The study imaged experimental stone tools in the AFM and from these images determined the average roughness values for flint surfaces’ peaks and valleys before and after different types of polishes.

The importance of this initial study is manifest in subsequent pilot programs with optical interferometry (Anderson *et al.* 1998; Gonzalez-Urquijo and Ibanez-Estevez 2003) tribology/vertical-scanning interferometer (Anderson *et al.* 2006), laser profilometry (Stemp and Stemp 2001, 2003; Stemp *et al.* 2010), and laser scanning confocal microscopy (Evans and Donahue 2008). With the exception of the study by Anderson *et al.* (2006) of a single class of plant threshing tools, none of these programs were able to analyze actual archaeological specimens. And none of these studies produced more accurate quantitative measures of microwear polish differentiation that Kimball *et al.* (1995, 1998). However, this initial study was constrained by the size restriction of the old AFM stage, which necessitated the use of small (<2 cm) replica tools, and thus prevented the expansion of the findings to actual archaeological specimens. The AFM used in this study is a Veeco Icon which has a large sample stage that can accommodate tools up to several centimeters in height and up to 20 x 20 cm in size, which allows us to image real artifacts without cutting them to fit to a small sample holder.

In our study, we combine the Keeley method with AFM to analyze six different polishes (meat, bone, fresh hide, dry hide, hafting, and wood) on actual archeological artifacts as opposed to experimental stone tools. The Keeley method is ideal for first identifying the locations and types of the wear traces. However, it is based on qualitative-based attributes and does not provide quantitative data and the magnification of typical incident-light binocular microscopes is relatively low. AFM can provide high magnification imaging and quantitative information about the polish topography. However, low magnification images are not always possible as scan head is controlled by a piezo which limits scan sizes to a maximum of roughly 0.1 mm x 0.1 mm. Also, the piezo limits the

heights of the features in the scan that can be imaged to 20 microns or less, depending on the microscope. This means that many use traces cannot be imaged in the AFM, as the tool is simply too rough.

The artifacts studied herein are Mousterian (i.e., Middle Palaeolithic technology of Neanderthals – see Bordes 1968) stone tools from Myshtulagty Lagat (Weasel Cave), North Ossetia, Russia (Fig. 6). The cave is at 1125 m AMSL in the north-central Caucasus Mountains (Hidjrati *et al.* 2003, 2009). Preservation is exceptional including carbonized seeds, nuts and wood, and the excellent preservation of microwear polishes on all flint artifacts. Excavations by N. Hidjrati since 1981 reveal an intact stratigraphy of over 22 vertical meters representing 36 distinct layers (Fig. 7). As of 2010, 23 distinct layers can be assigned to the Middle Paleolithic, and contain Typical Mousterian or Denticulate Mousterian with Levallois blades industries. They span the Middle to Late Pleistocene with Layer 14 dated to Isotope Stage 5e. The tools analyzed in this study represent only five tools of the 20 Mousterian flint being studied. (All 94 flint artifacts, including debitage) have been analyzed by the Keeley Method, which represents 100% of the flint tools excavated to date from Weasel Cave. And a sample of 178 quartzite artifacts, of the thousands recovered, have been analyzed as well. A summary description of these analyses are reported in Hidjrati *et al.* (in press). The five Mousterian tools derive from Layers 12-13, which date to between 50,000 and 90,000 (Isotope Stages 4-5c) based upon pollen and microtine studies. These tools are of Ossetian flint from sources ~20-30 km due west and south of Weasel Cave. It is a very fine-grained and the surfaces appear fresh.



Fig 6. Map showing Myshtulagty Lagat (Weasel Cave) in North Ossetia, Russia.

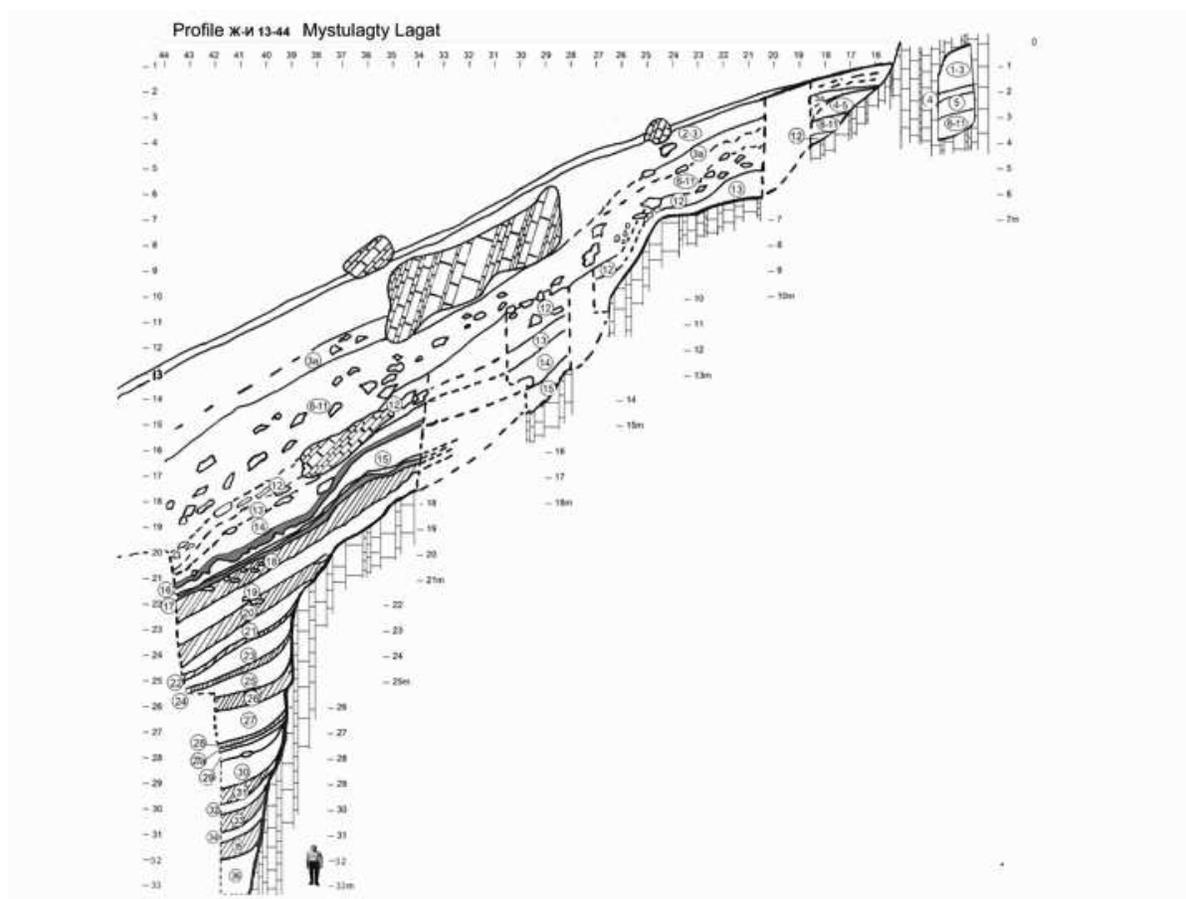


Fig 7. Excavations from Weasel Cave reveal an intact stratigraphy of over 22 vertical meters representing 36 distinct layers.

EXPERIMENTAL TECHNIQUES

After excavation, the flint and quartzite artifacts from Weasel Cave are cleaned in a weak HCl bath to remove the calcite coating on almost all specimens. The tools were then ultrasonically cleaned in an ammonia detergent bath before inspection for microwear traces with an Olympus BH incident-light microscope under 50, 100, and 200x. The observed polishes conform to those found on experimental tools by Kimball as well as those expected by a large number of microwear analysts employing this same method of cleaning and

microscopy. Each polish is described according to the formal Keeley-Plisson-Kimball schema (Kimball 1989).

Once the polishes were identified via the Keeley method, they were mounted on clay for imaging in the Veeco Icon AFM. The Veeco Icon has an optical scope mounted above the sample stage, so that the polishes can be precisely positioned for proper imaging. The samples were imaged in tapping mode. In tapping mode AFM, a tip mounted on a cantilever oscillates at high frequency with constant amplitude. The tip is approached towards the sample until the amplitude of oscillation is damped due to the tip tapping on the sample surface. This constant amplitude of oscillation is maintained by moving the tip up and down in response to surface topography as the tip is scanned back and forth across the sample surface. The motion of the tip is controlled by a piezo in a feedback loop with the control electronics. Tapping mode AFM was utilized as opposed to contact mode AFM to ensure that the wear to the AFM tip was minimal; tip wear can limit the accuracy of the roughness measurements. Our AFM tips and cantilevers are commercially available Veeco probes with resonance frequencies of 190 kHz and tip radii of less than 10 nm. The tip was changed frequently to ensure accurate roughness measurements. The AFM images shown have been flattened to correct for the tilt of the sample plane, but have otherwise not been filtered or enhanced. All of the AFM images were acquired at the same image size, 50 x 50 micron, and 512 x 512 pixels, at a scan rate of 0.15 Hz.

We acquired AFM images of the use trace regions and also of control areas that had no evidence of wear. We used the AFM software to calculate the average roughness of the peaks, valleys, and transition regions in each image. We used ~10 different 2 x 2 μm^2 square areas of each type to perform this analysis, predicting that the peaks, valleys, and slopes or

transitions should be worn at different rates. The results and corresponding uncertainties are displayed in tabular format. We report the values of the average roughness, skewness, and excess kurtosis for the 50 micron x 50 micron AFM images. Skewness (Sk) is a measure of the degree of symmetry of the image; images with a Gaussian distribution of surface roughness values have a skewness of zero. Images with plateaus have lower values of skewness than images with isolated steep peaks.

$$Sk = \frac{1}{nR_q} \sum_{i=1}^n (Z_i - \bar{Z})^3 \quad (2)$$

Here, n is the number of data points, R_q is the root mean square roughness of the image, Z_i is the height of the i^{th} data point, and \bar{Z} is the average height of the image. Kurtosis (K) shows the pointedness or bluntness of the distribution of the roughness values. Smoother profiles have less variation in their roughness values, and therefore the distribution of roughness values is more narrow than a Gaussian distribution. This causes the kurtosis for more uniform surfaces to be higher than surfaces with greater variation in roughness values.

$$K = \left[\frac{1}{nR_q^2} \sum_{i=1}^n (Z_i - \bar{Z})^4 \right] - 3 \quad (3)$$

RESULTS AND ANALYSIS

Wood-working polish

Burin WC-1096/1097 is interpreted to have been used to plane wood along three burinated edges, but was not hafted. The scanned Use-Trace 1c is indicated by the square in Figure 8. Digital microphotographs of the microwear polish at 200x is shown in Figure 9

(the digital image is 344 um in height). The identification of this microwear trace as wood polish is based upon an invasive to spreading *extent*, smooth, united *texture*, a fluid polish with a *coalescence* following the entire microtopography. The *polish* is most pronounced at the higher elevations, and continues over the edge rather than rounding or otherwise significantly modifying it. The *contour* of the polish is irregularly clear. It is a *very bright* polish rarely with striations.

The AFM images were acquired of the polish as well as an adjacent unmodified surface thus representing a control for this tool (Fig. 10 and Fig. 11). (It is important to keep in mind that there is some variation in the graininess of these Mousterian tools as they are on different types of Ossetian flints, presently unsourced.) The microwear polish (Fig. 10) is scanned over a 50 x 50 micron. The edge of the tool is close to the location of the area imaged to the right of the image. The control image (Fig. 11) has been cropped to 50 x 45 micron due to streaking in the AFM image caused by the extremely rough surface of the tool. It is easy to see the areas of microwear in these top view scans. The peaks of the surface have been almost completely worn away and flattened smooth. The valleys remain rough, with no evidence of filling in with wear debris. This is reflected in the analysis of the roughness data from the peaks, valleys, and transition regions, as shown in Table I. The roughness, skewness and kurtosis as determined from the AFM scans is also included in Table II.

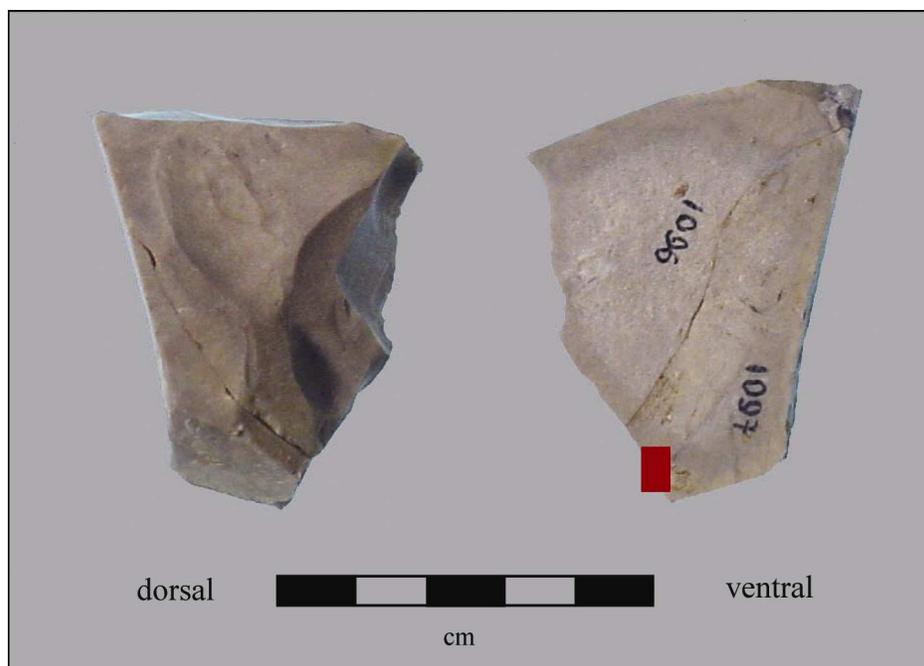


Fig 8. Use-Trace 1c on Burin WC-1096/1097 is indicated by the box.



Fig 9. 200x optical image of the wood-working polish, with the AFM scan indicated by the arrow.

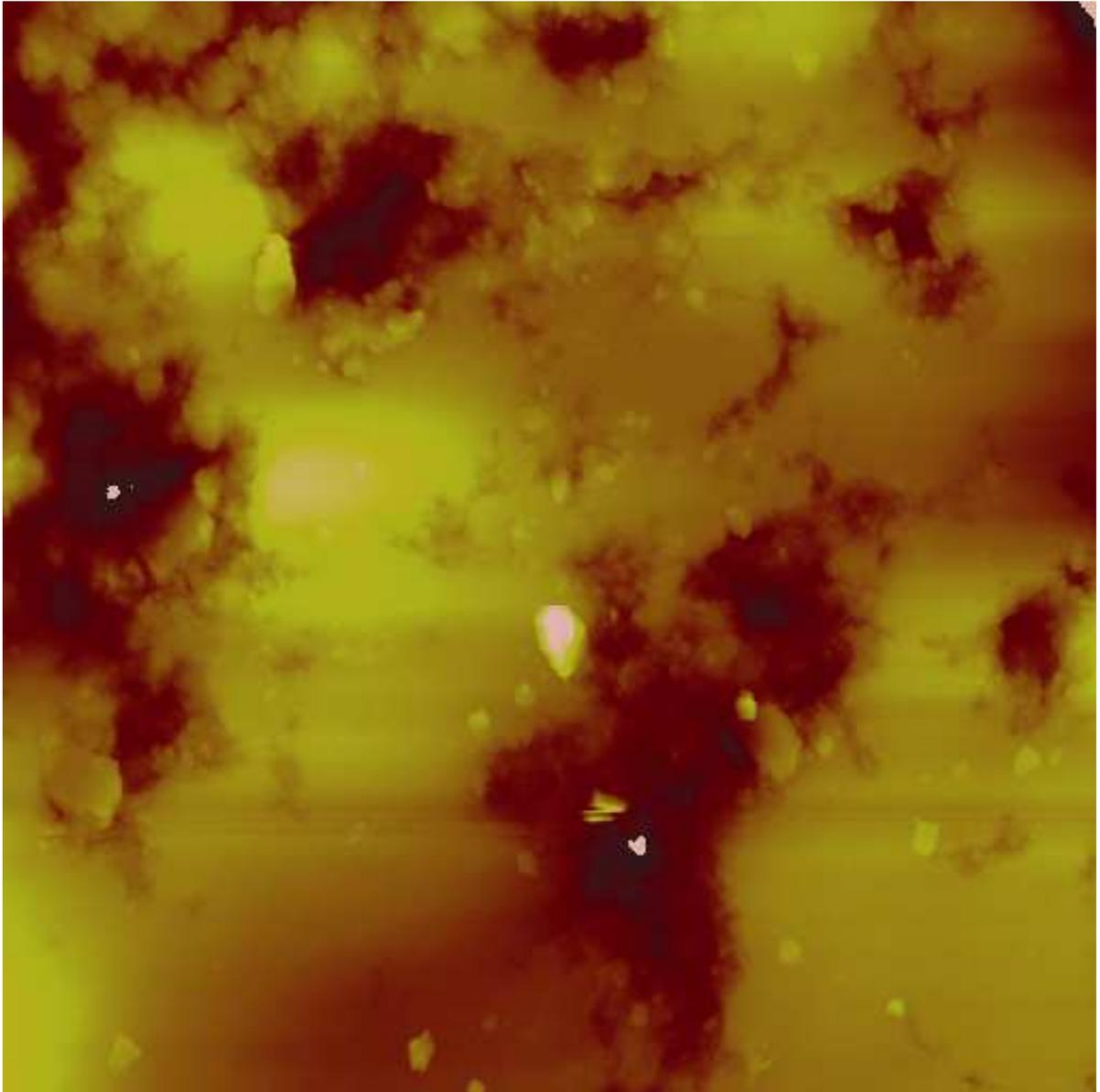


Fig 10. AFM scan of the wood-working polish region. Notice the smooth texture across much of the polish.

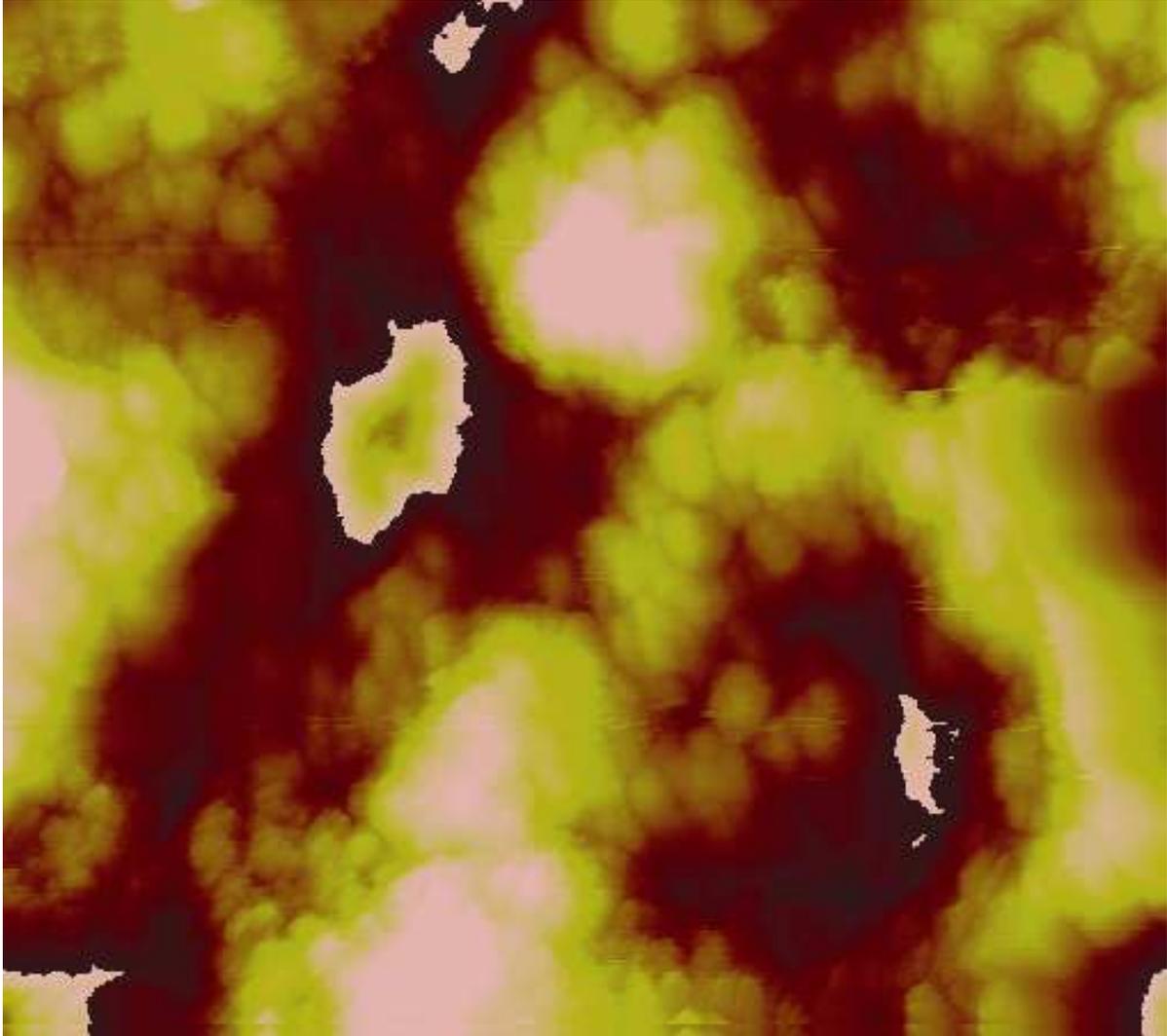


Fig 11. AFM scan of an adjacent unmodified region, representing a control for the artifact.

Fresh hide-working polish

Atypical Mousterian Point WC-39/1988 (Fig. 12) is interpreted to have been used to clean fresh hide (at the distal end) and whittling wood (along the lateral right edge) in a hafted mode. Use-trace 4b is illustrated here, was used to clean hide in a fresh state – that is, to remove adhering tissues on the interior hide surface. Digital microphotographs of the

microwear polish at 200x is shown in Figure 13 (the digital image is 344 um in height). The identification of this microwear trace as fresh hide-working polish is based upon a polish is fluid and grainy following the microtopography. With intensive work, the polish modifies the higher portion of the microtopography more significantly. It's extent is invasive and exhibits an average texture. The contour of the polish is fuzzy and exhibits an average or "matte" brightness. Striations are present and are short, wide, and deep into the fresh hide polish.

AFM scans were acquired of the microwear trace (Fig. 14) and a control spot (Fig. 15) over an areas of 50 x 50 microns. The edge of the tool is close to the location of the wear trace area imaged, and is to the right of the image. The AFM image of the microwear trace gives information about the polish morphology that is not easily seen in the optical image. Note the striations and directionality of the wear; the observable striations within the microwear polish vary from being parallel to at a small angle from the working edge, thus indicating the tool kinematics. This is indicative of a cutting motion of the tool through the tissues attached to the hide. Quantitative statistics on these images are shown in Tables I and II.

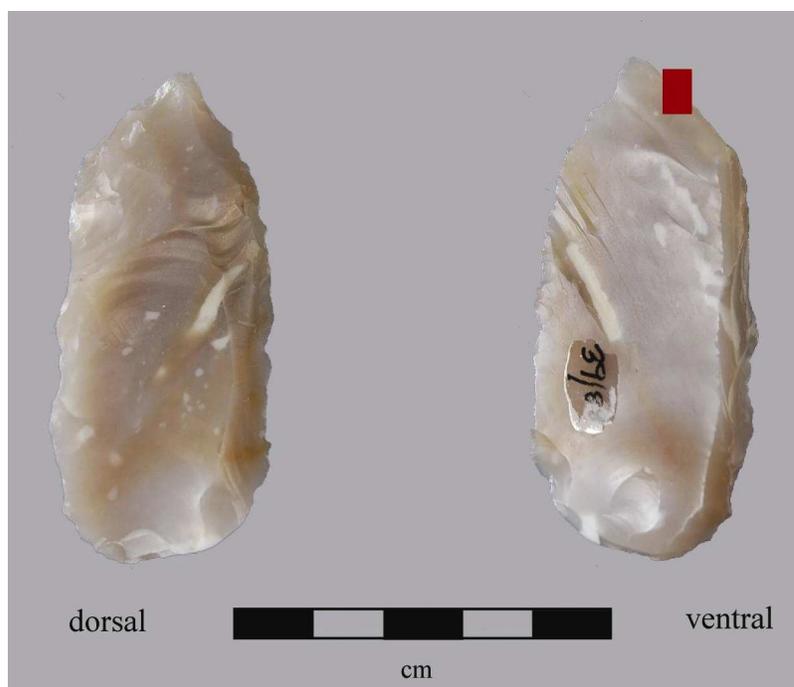


Fig 12. Mousterian Point WC-39/1988 with use-trace 4b indicated by the box.



Fig 13. 200x optical image of the fresh hide-working polish. The AFM scan was performed in the central smooth region along the edge of the polish.

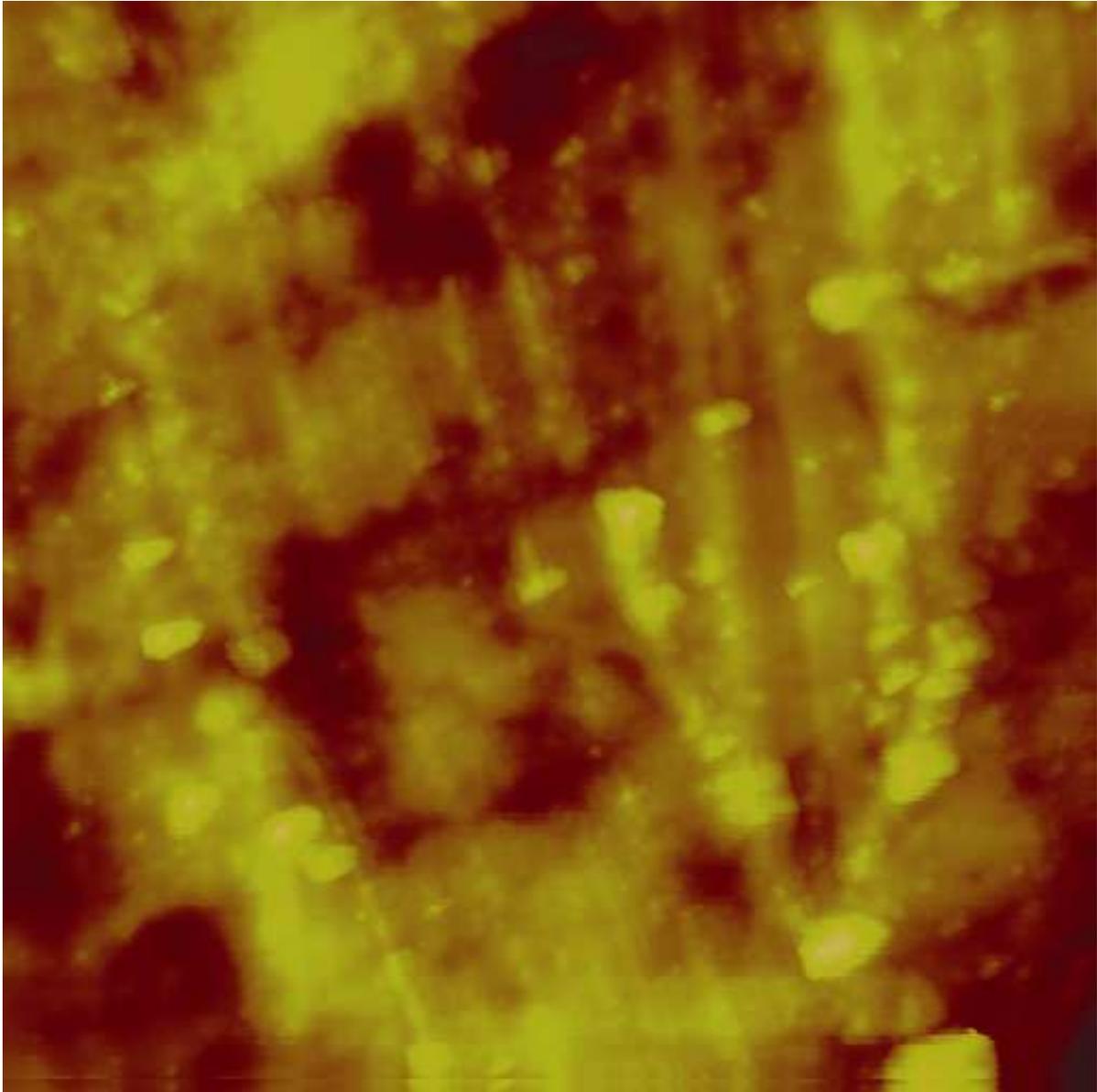


Fig 14. AFM scan of the fresh hide-working polish. The edge of the tool is located immediately to the right of the image. The directionality of the striations indicates a cutting motion of the tool through the fresh-hide.

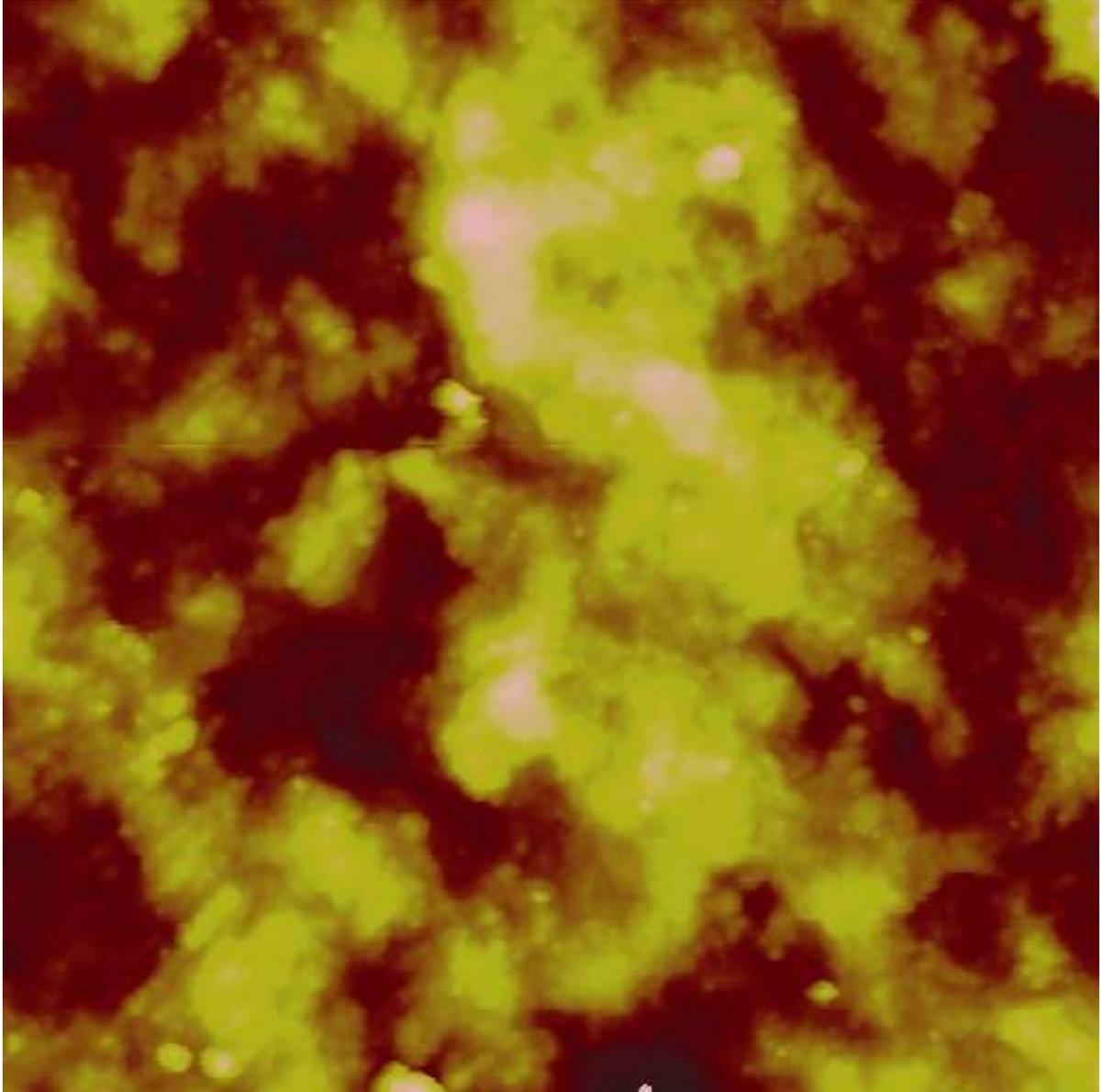


Fig 15. AFM scan of an unmodified control region adjacent to the fresh hide-working polish.

Dry hide-working polish

Mousterian Point WC-306/1988 is interpreted to have been used in two functions: (1) butchery along the lateral right edge; and (2) planing wood along the lateral left edge. The

illustrated use-trace 3a (Fig. 16) exhibits dry hide polish in this location (along with microwear polishes from cutting through the hide in fresh condition, cutting through meat, and in one place contacting bone. Digital microphotographs of the microwear polish at 200x is shown in Figure 17 (the digital image is 344 um in height). This is the classic manifestation of heavy butchery (Kimball 1989; Yerkes 1987,1994), as opposed to simply cutting through meaty tissues. In this case, it appears that processing of the carcass continued until the hide was relatively dry. The illustrated dry hide polish is defined as a soft, grainy polish with significant edge rounding and modification of the microtopography. The texture is dense, the contour is fuzzy, and exhibits a matte/weak brightness. The numerous striations are long, wide, and deep. The extent of the polish is moderate. The image also shows that abrasive wear of the tool edge caused a rounding and smoothing of the working edge of the tool.

AFM images were acquired of the microwear polish (Fig. 18) and an unused control location nearby (Fig. 19). Both images are 50 x 50 um. The edge of the tool to the right of the imaged top view. Quantitative statistics on these images are shown in Tables I and II.

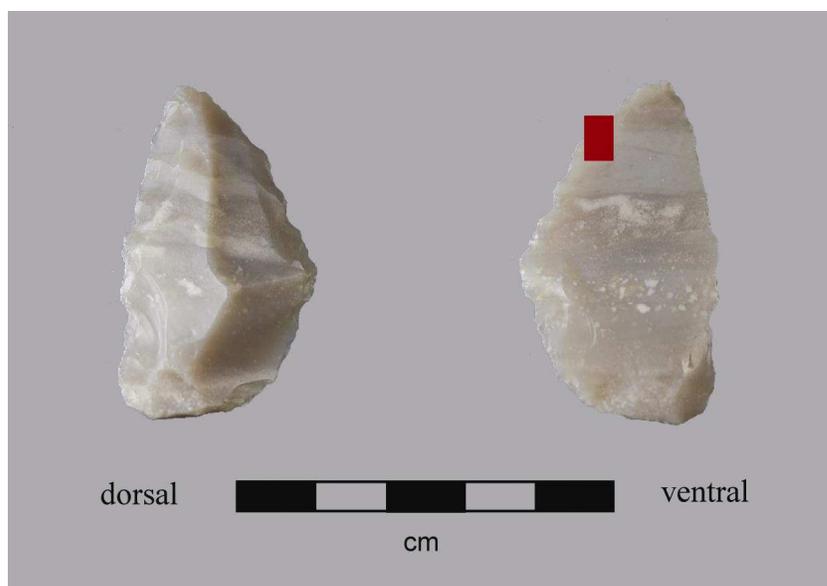


Fig 16. Mousterian Point WC-306/1988 with use-trace 3a indicated by the box on the ventral aspect.



Fig 17. 200x optical image of the dry hide-working polish. The AFM scan was performed near the dull, rounded edge of the tool.

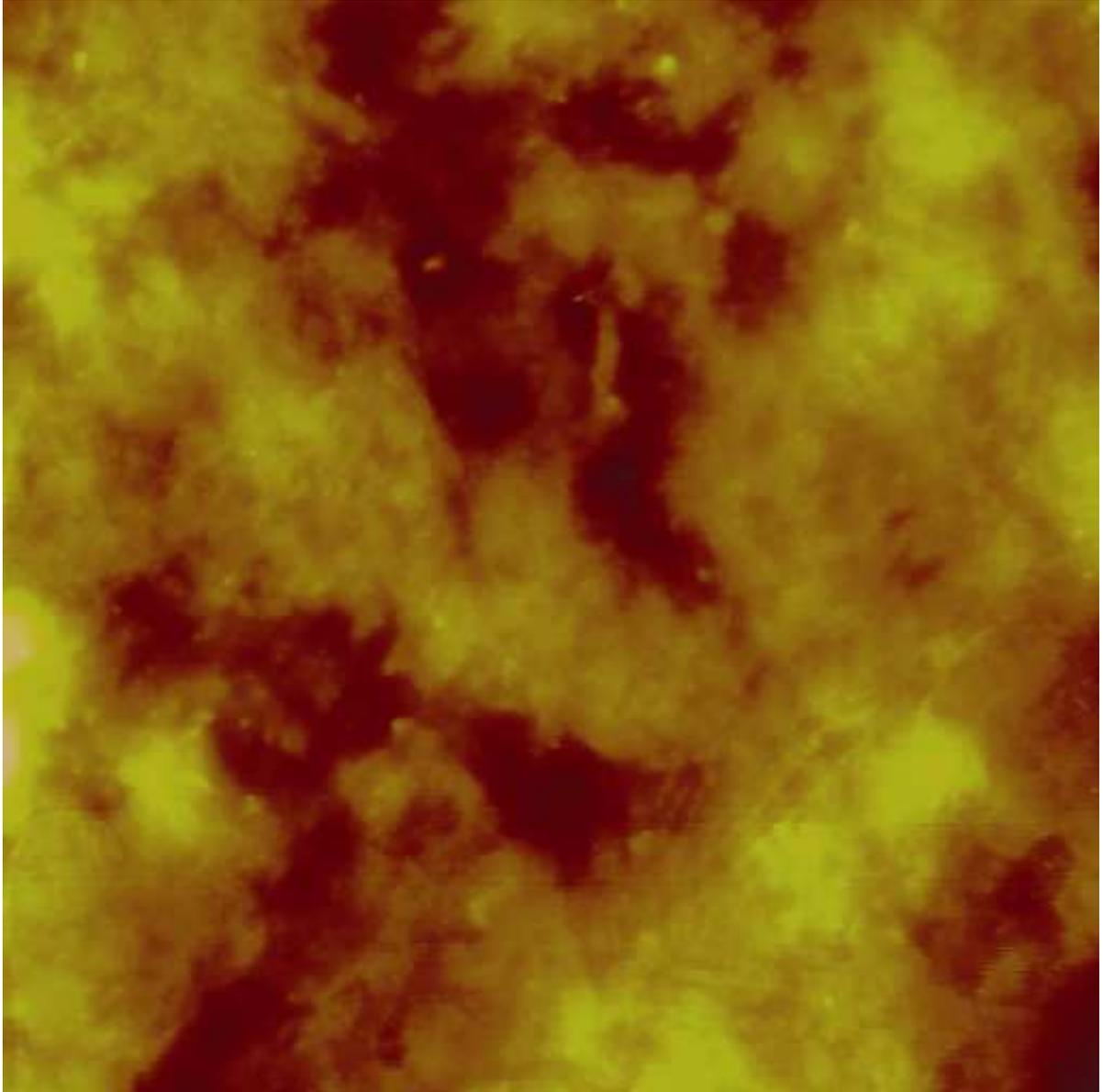


Fig 18. AFM scan of the dry hide-working polish. The edge of the tool is located to the right of the image.

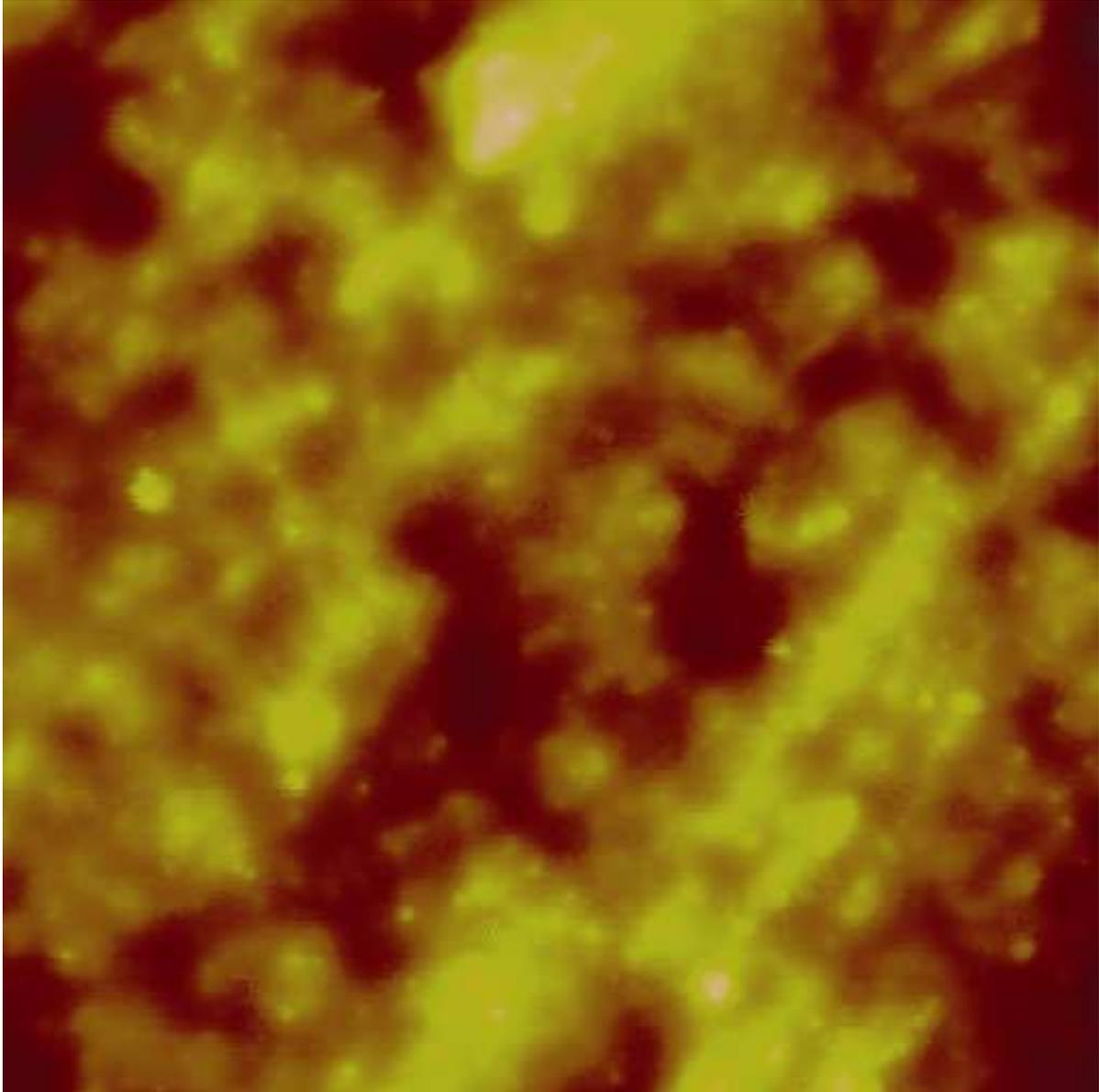


Fig 19. AFM scan of an unmodified control region adjacent to the dry hide-working polish.

Meat cutting polish

Levallois Blade WC-1047/1988 (Fig. 20) is interpreted to have been used in butchery (lateral right edge) and wood planing (proximal edge). Use-trace 6b is identified as a polish

from cutting meat cutting, but all along this working edge were meat and fresh hide polishes. The identification of this microwear trace as meat is defined by a very fluid polish which affects the entire microtopography alike without major alteration of the relief. The texture is dense and the contour is fuzzy, but more evident at 50x-100x (see Fig. 21). Yet, it forms a continuous linear band of polish along the working edge. The brightness is average and somewhat lustrous. A few striations are evident and they are narrow, straight, and short; and indicate the direction of tool use.

AFM images were acquired of the micropolish (Fig. 22) and an unused control location (Fig. 23), and are 50 x 50 um. Quantitative statistics on these images are shown in Tables I and II. The edge of the tool is to the right of the image. The AFM image of the wear spot gives information about the polish morphology that is not so easily seen in the optical image – striations which indicate the directionality in the polish. There are two distinct striae, running both parallel (top of image) and oblique (bottom of image) to the edge. It seems as though the tool was used in a sawing motion, which would give striations parallel to the edge, and then later more in a slicing or scraping motion, giving oblique striations relative to the edge.



Fig 20. Levallois Blade WC-1047/1988 with use-trace 6b indicated by the box.



Fig 21. Optical image of the meat cutting polish. The AFM scan was performed near the interior working edge.

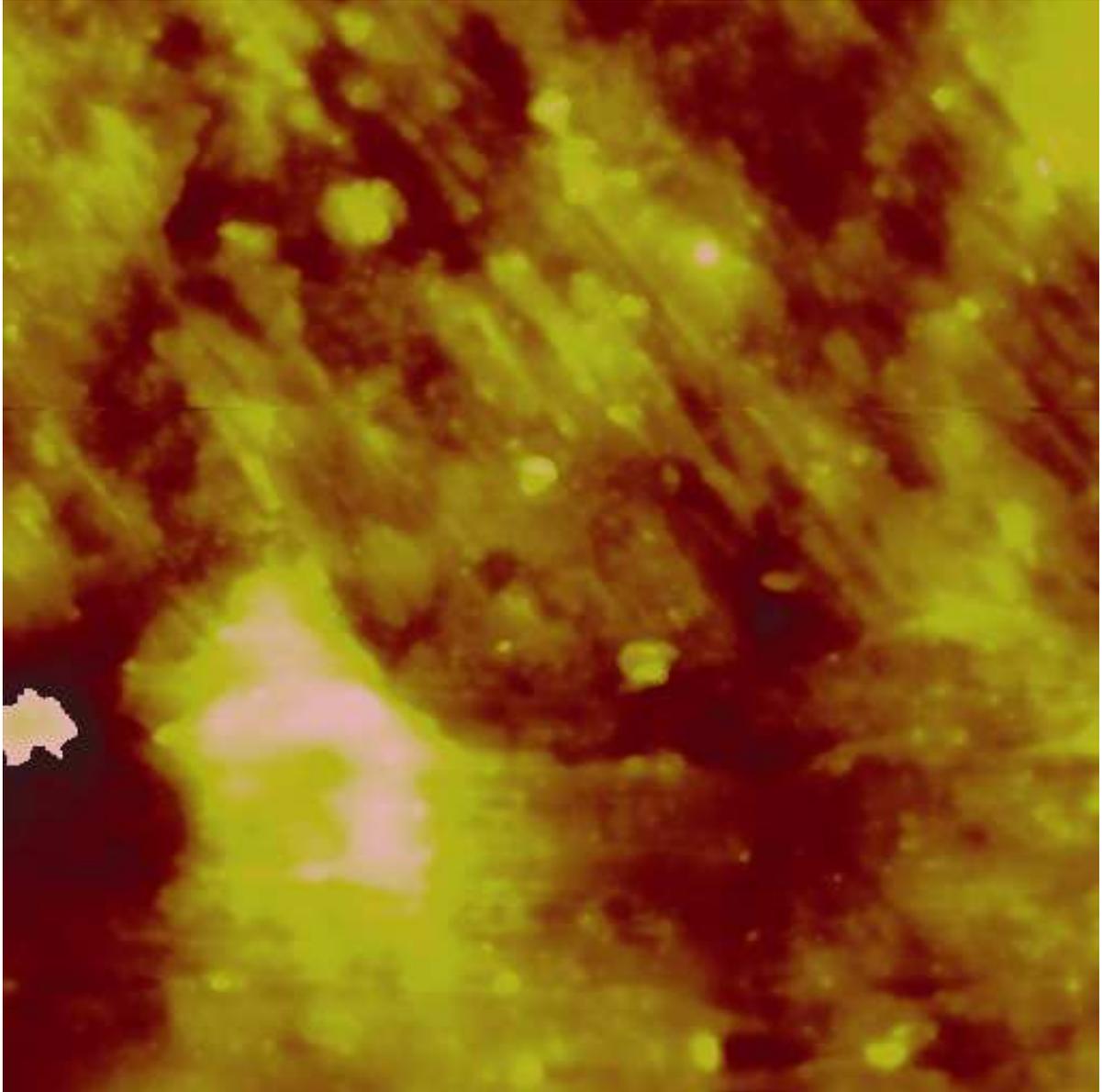


Fig 22. AFM scan of the meat cutting polish. The working edge of the tool is located to the right of the image.

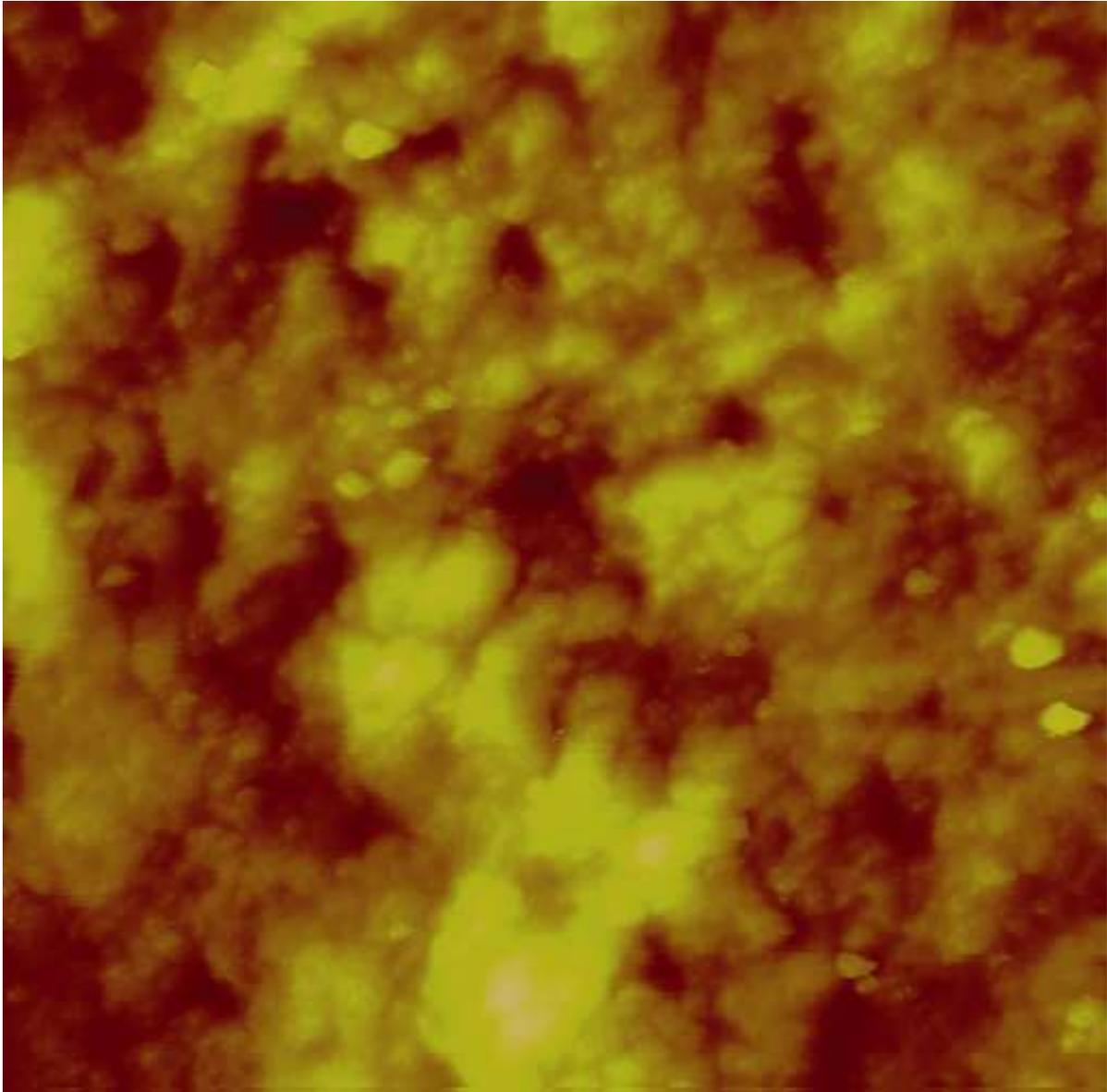


Fig 23. AFM scan of an unmodified control region adjacent to the meat cutting polish.

Bone polish

Atypical Levallois Point WC-520 (Fig. 24) was observed to have been used in butchery (both lateral edges) in a hafted mode. This is indicated by the presence of fresh hide

and meat polishes continuously distributed along the working edge, and the occasional spots of bone polish. At the illustrated location a spot of bone polish was observed at the distal lateral left point (Fig. 25). Elsewhere along both lateral edges, polishes from cutting through fresh hide and meat were observed. At an isolated location, bone polish is a hard, undulating and bright polish that appears to be spreading. Keeley (1980) refers to this characteristic of the coalescence as a “melted snow” appearance. Bone polish concentrates at high elevations of the microtopography and at projections along the edge. The contour is clear and rather abrupt. Bone polish is observed experimentally to have an isolated extent in sawing/cutting actions. The texture is dense.

AFM images were acquired for 50 x 50 um areas (Fig. 26 and Fig. 27). Quantitative statistics on these images are shown in Tables I and II. The edge of the tool is to the right of the image. Bone polish is similar to the wood polish in that the microwear is much more abrasive, resulting in a polish that is very smooth and flat. However, this example of bone polish has a distinct directionality to small striations (wear tracks), oriented oblique to the edge. This indicates that the tool was used in a slicing or hacking motion into the bone, as one would expect during butchery.



Fig 24. Atypical Levallois Point WC-520 with the region of interest indicated by the square.



Fig 25. Optical image of the bone polish. The AFM scan was performed in the smooth region near the working edge of the tool.

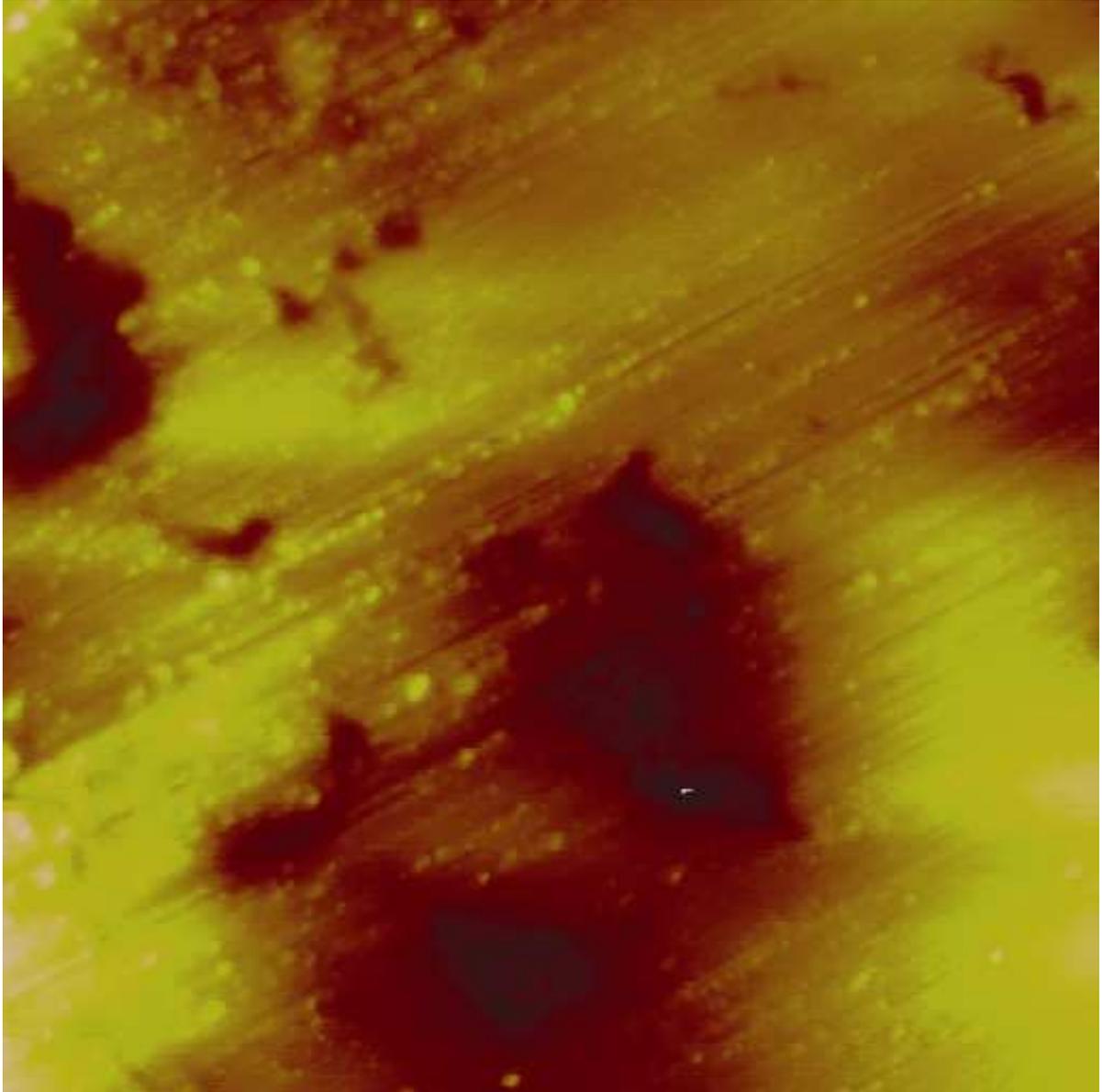


Fig 26. AFM scan of the bone polish. The edge of the tool is located to the left of the image.

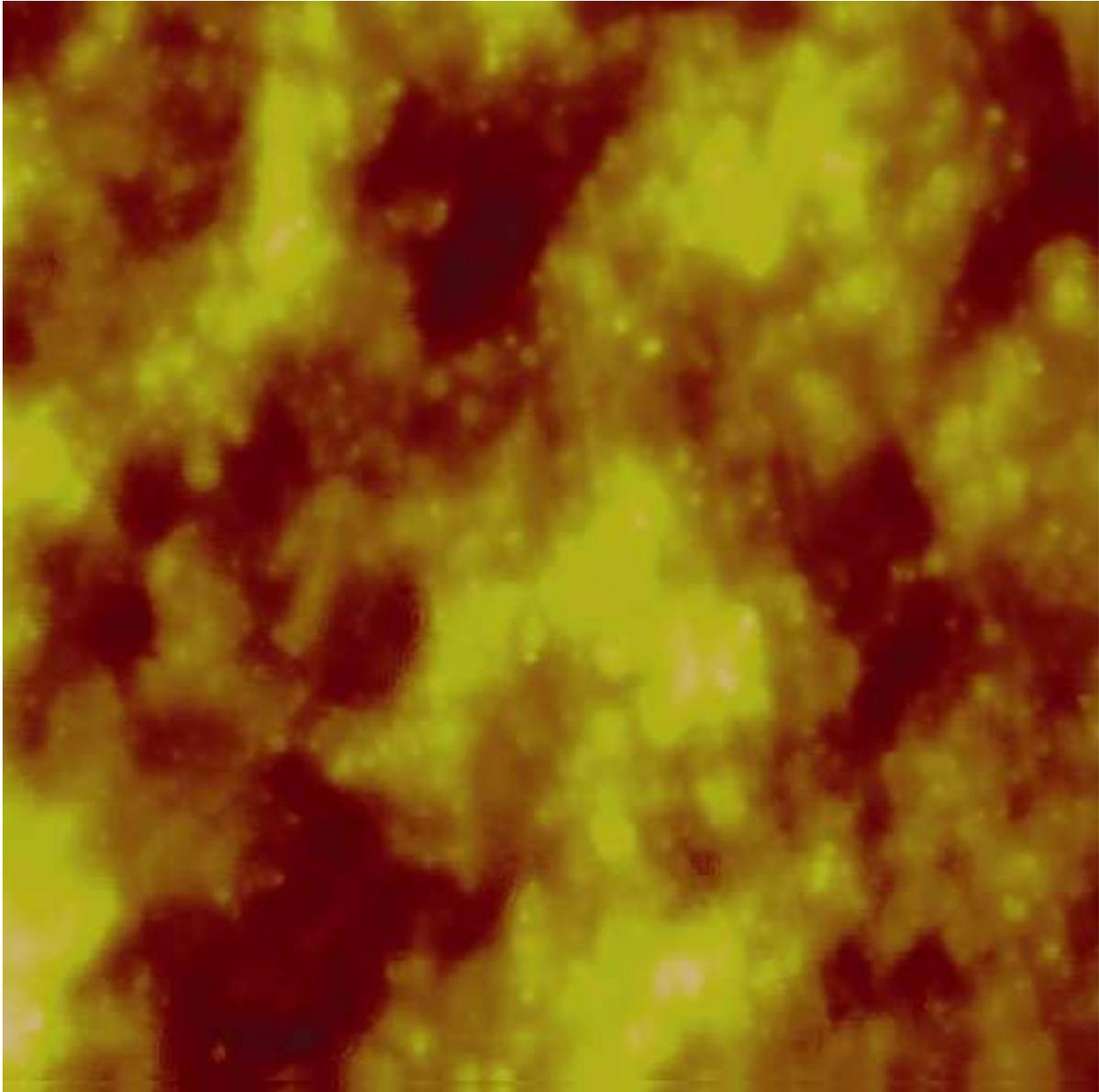


Fig 27. AFM scan of an unmodified control region adjacent to the bone polish.

Hafting trace polish

Some stone tools, such as Mousterian Point WC-306/1988 pictured in Figure 16, contain hafting traces, evidence of wear created by the positioning of the tool in a wood or

bone handle. They are evidenced by a very smooth, brilliant polish, striations, and modifications of the tool edge by micro-scarring or edge-rounding (Rots 2008, 2010). When viewed at the correct angle a hafting trace can be optically reflective. Dominant variables in the formation of hafting traces are the type of use, the hafting material, and the hafting arrangement. The coarseness and morphology of the tool prior to use are secondary variables in determining the characteristics of the polish. The optical image (Fig. 28) shows hafting trace 1e on the dorsal side of the tool near the prominent dorsal ridge at 100x magnification. The wear spot is very reflective and exhibits directionality of the polish.

AFM images of the hafting trace (Fig. 29) and the control spot (Fig. 30) are shown below. Both images are 50 micron x 50 micron and have z-scale bars on the right side. While the control image is topographically unaltered, the hafting trace shows visible wear scars with directionality. This is significant in identifying the type of wear spot because the edge of the tool is just above the top of the image. The wear scars are parallel to the edge of the tool, a common evidence for characterizing hafting traces. Quantitative statistics on these images are shown in Tables I and II.

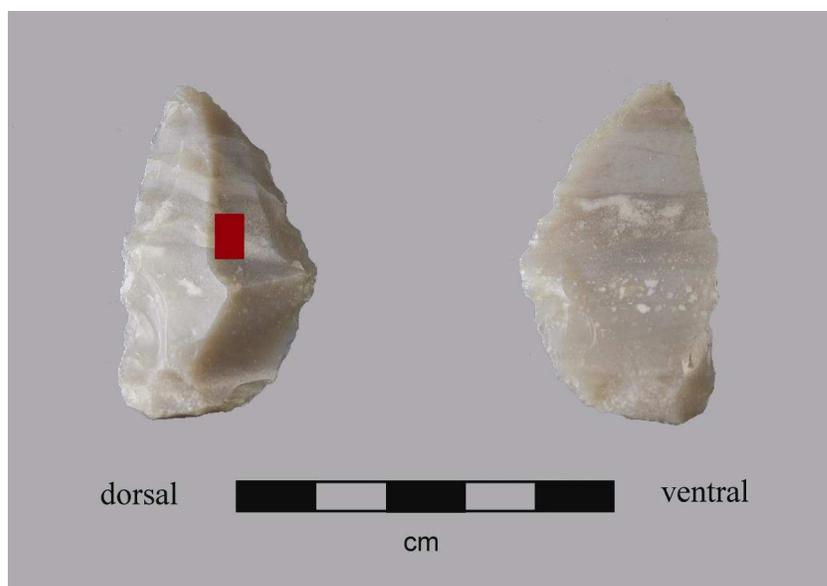


Fig 28. 100x optical image of hafting trace 1e on Mousterian Point WC-306/1988. The AFM scan was taken in the center of the polish.

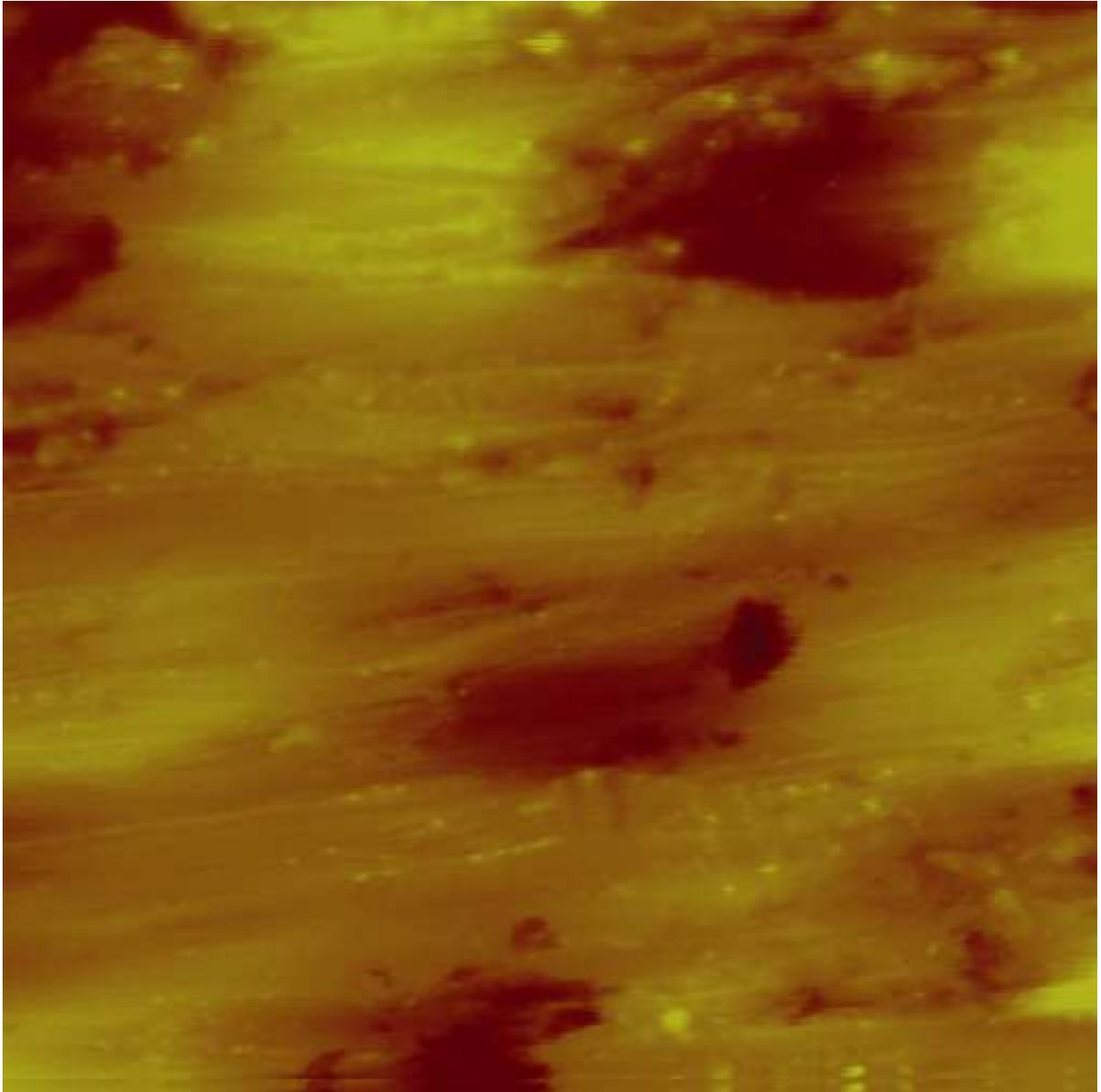


Fig 29. AFM scan of the hafting trace. The dorsal ridge of the tool is located to the left of the image.

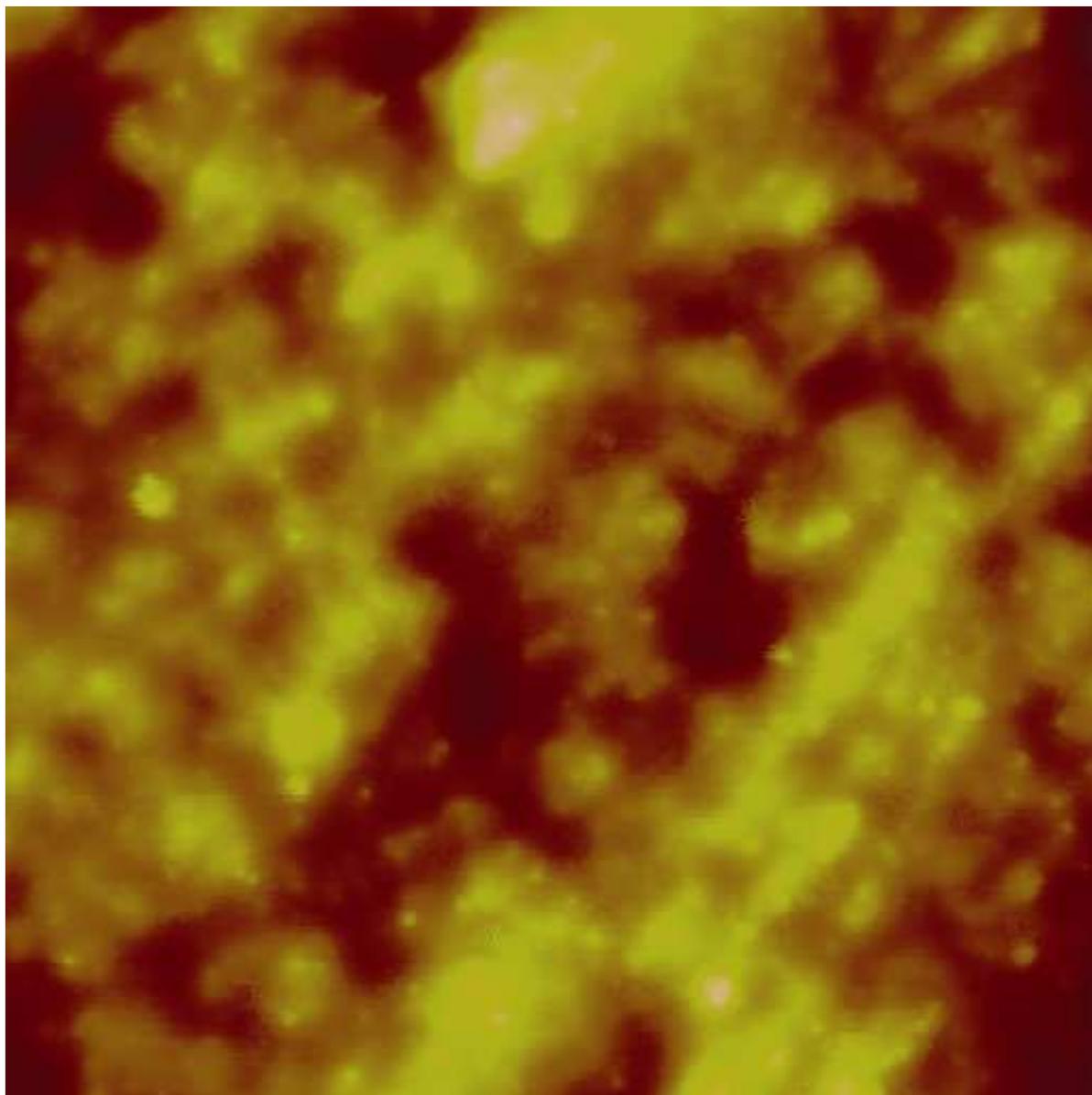


Fig 30. AFM scan of an unmodified control region adjacent to the hafting trace.

Table I. Average roughness (R_a) and uncertainties for the peaks, valleys, and transition regions of the various polishes, as determined by AFM.

Polish Type	R_a (nm) Peaks	R_a (nm) Valleys	R_a (nm) Transitions
Wood Working	8 ± 2	113 ± 9	40 ± 5
Wood Control	52 ± 7	70 ± 9	56 ± 6
Fresh Hide Working	19 ± 3	33 ± 2	22 ± 3
Fresh Hide Control	38 ± 5	54 ± 8	45 ± 5
Dry Hide Working	24 ± 3	31 ± 3	25 ± 3
Dry Hide Control	27 ± 4	26 ± 3	26 ± 3
Meat Cutting	33 ± 3	38 ± 6	29 ± 5
Meat Control	41 ± 4	51 ± 6	26 ± 4
Bone Working	12 ± 2	23 ± 3	12 ± 2
Bone Control	23 ± 3	28 ± 3	25 ± 3
Hafting Trace	18 ± 4	21 ± 3	11 ± 2
Hafting Control	27 ± 4	26 ± 4	26 ± 3

Table II. Average roughness (R_a), skewness (Sk), and kurtosis (K) of various polishes and their controls from AFM image analysis of 50 micron x 50 micron area. Due to the streaking in the AFM control image of the wood polish tool, this tool was analyzed in both control and polish image for a 44 micron x 44 micron area.

Polish type	R_a (nm)	Sk	K
Wood	200	-0.027	0.69
Wood Control	622	0.093	0.49
Fresh Hide	169	-0.361	0.28
Fresh Hide Control	274	0.192	-0.52
Dry Hide	185	-0.340	0.10
Dry Hide Control	180	0.008	0.03
Meat	214	0.227	1.78
Meat Control	157	0.446	0.08
Bone	110	-0.438	0.30
Bone Control	189	0.142	-0.10
Hafting Trace	105	-0.324	0.72
Hafting Control	180	0.008	0.03

CONCLUSIONS

A previous AFM study on experimental flint tools (Kimball *et al.* 1995) showed that the surface roughness was lowest for tools used in working antler, followed closely by wood working, then dry hide working, and finally meat cutting was the least developed polish. Our AFM study on Neanderthal stone tools closely agrees with this observation. The roughness of the peaks as determined by AFM for the bone working and wood working polishes are the same within the uncertainties, followed by fresh hide, dry hide and then meat. In the Mansur-Franchomme (1983) study on experimental stone tools, it was noted that when there is moisture present during use the wear is more extreme and less localized to the peaks. This

agrees with our results for differentiating microwear polishes from working animal hides in fresh and dry states. Studying the morphology from the images of the various techniques, it can be seen that the wear is more uniform and widespread for the fresh hide working than for the dry hide working.

For all of the traces, the skewness determined from the AFM images is reduced for the polish vs. control regions. Since the skewness is lower for surfaces with more plateaus and higher for surfaces with isolated steep peaks, lower values of skewness imply a more worn surface. For all of the traces, the excess kurtosis determined from the AFM images is increased for the polish vs. control regions. An increase in kurtosis implies a more uniform surface roughness. Skewness and kurtosis are therefore parameters that can help give a quantitative measure to distinguish use traces for both experimental tools and archeological artifacts. It is exciting that the less obvious polishes, such as meat or dry hide, show quantitative differences in these parameters, as these polishes are difficult to verify qualitatively by simply viewing the morphology or examining the roughness alone.

In conclusion, the AFM can provide useful quantitative information in the study of microwear polishes of archeological artifacts at the very small scale. As opposed to other micro and nanoscale techniques, such as scanning electron microscopy, it can provide quantitative roughness analysis without sputter coating the artifact or casting. THE AFM can also provide higher resolution images than optical interferometry or laser scanning confocal microscopy, techniques commonly used for quantitative analysis of lithic artifacts. We are currently pursuing more studies with experimental stone tools made from the types of flints used by these Neanderthals, in order to better compare known stone tool function and duration of use for experimental tools vs. artifacts. This is necessary because it has been

shown that the polish development can vary greatly depending on the properties of the lithic raw material. Future works will also show optical interferometry analysis of the artifacts.

The various techniques used to analyze stone tools such as AFM, SEM, the Keeley Method, optical interferometry, and laser scanning confocal microscopy, have their own advantages and disadvantages, and different types of information that can be obtained using each technique. We therefore believe it is important to use multiple analysis methods when a truly comprehensive microwear study is desired.

The first experimentally-based, microscopic approach to stone tool function was developed by Russian archaeologist Sergei A. Semenov (1957; translated into English in 1964) using both stereo- and incident-light microscopy (Fig. 31). He was able to observe use-wear traces which, when compared with microwear traces on experimentally used replica tools, permitted the identification of the kind of material being worked and the kinematics (or specific tool holding and hafting positions and the specific motions) of the tool. Through the relation of observed microwear traces on archaeological specimens to the same on experimental tools, the actual function of individual tools could be ascertained. This was largely possible because Semenov recognized that use-wear polishes varied according to the type of material worked (i.e., wood, bone, antler, ivory, hide, soft plant, etc.). However, these microwear polishes were only observable using the incident-light microscope.

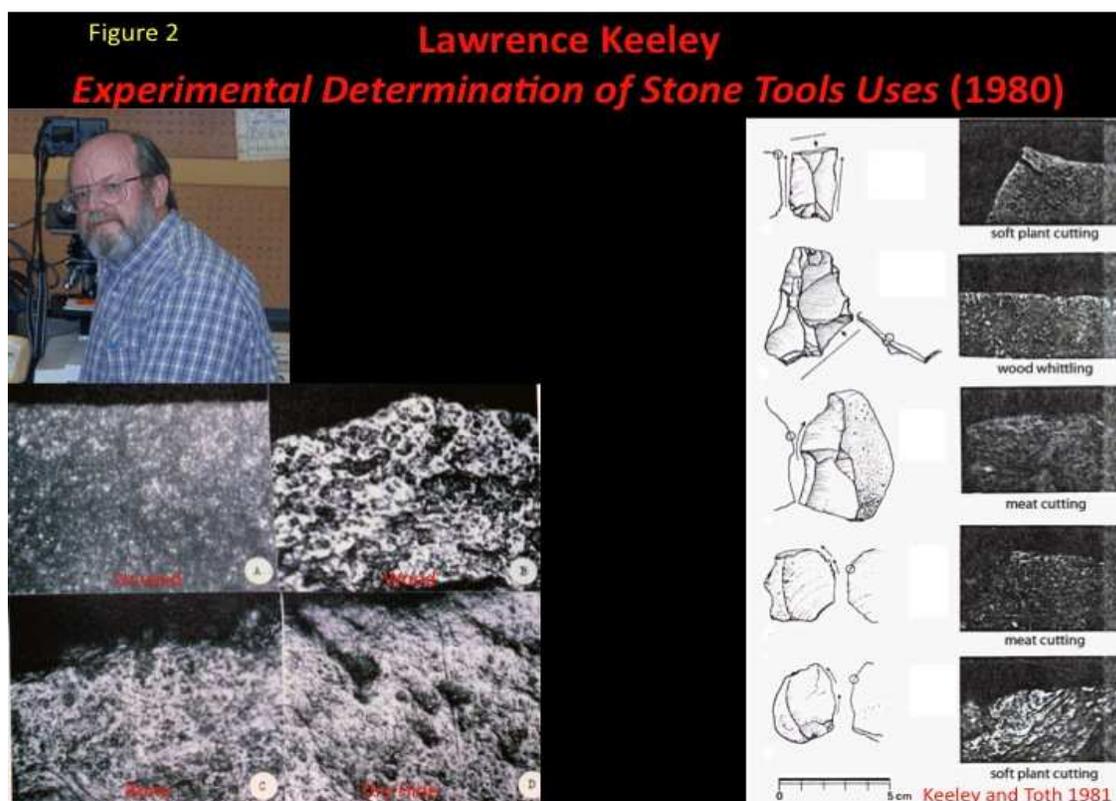


Fig 32. Microwear studies performed by Lawrence Keeley in 1980.

The first replication of Semenov's observations in the West was by the American Lawrence H. Keeley (1977, 1980) using the incident-light microscope with magnifications of 50x-400x (Fig. 32). The "Keeley Method" permitted the observation of additional microwear traces and a more refined characterization of the polishes. According to Keeley (1980) a microwear polish "can be described in terms of its brightness or dullness (that is, how much light it reflects) and its roughness or smoothness, as well as the presence of certain topographical features, like pits, undulations, and so forth." Thus, reflectivity, roughness, and microtopography are the major axes of variation expected for microwear polishes.



Fig 33. Low-magnification incident-light microscopy of microwear polishes on stone tools.

Accordingly, Keeley (1980) described polishes in qualitative terms: wood polish is very bright, very smooth in texture with a gently domed microtopography. Bone polish is bright with a micro-pitted texture (1980). Antler polish (Keeley 1980) is very bright and

smooth, but with gentle undulations (“melted snow” appearance) when well-developed. Polishes resulting from the cutting of soft plants are very bright, spreading, with fluid distribution along the microtopography and exhibits “comet-shaped pits” and “filled-in striations.” Polish resulting from cutting through meat is relatively brighter than the unaltered flint surface, its microtopography is clearly altered – “this luster seems to be the result of a smoothing of the microtopography on a very small scale. The normally grainy texture of a raw flint surface is replaced by a slight matt texture, which seems to preserve the very minute elevation and depression of the raw surface ...” (Keeley 1980). Hide-working polishes were observed to vary according to moisture and fat levels: fresh versus dry hide. In both cases, the entire microtopography is affected; but fresh hide polish appears “greasy” somewhat like meat polish, while dry hide polish exhibits a rough/matt/dull texture which increasing with work (Keeley 1980).

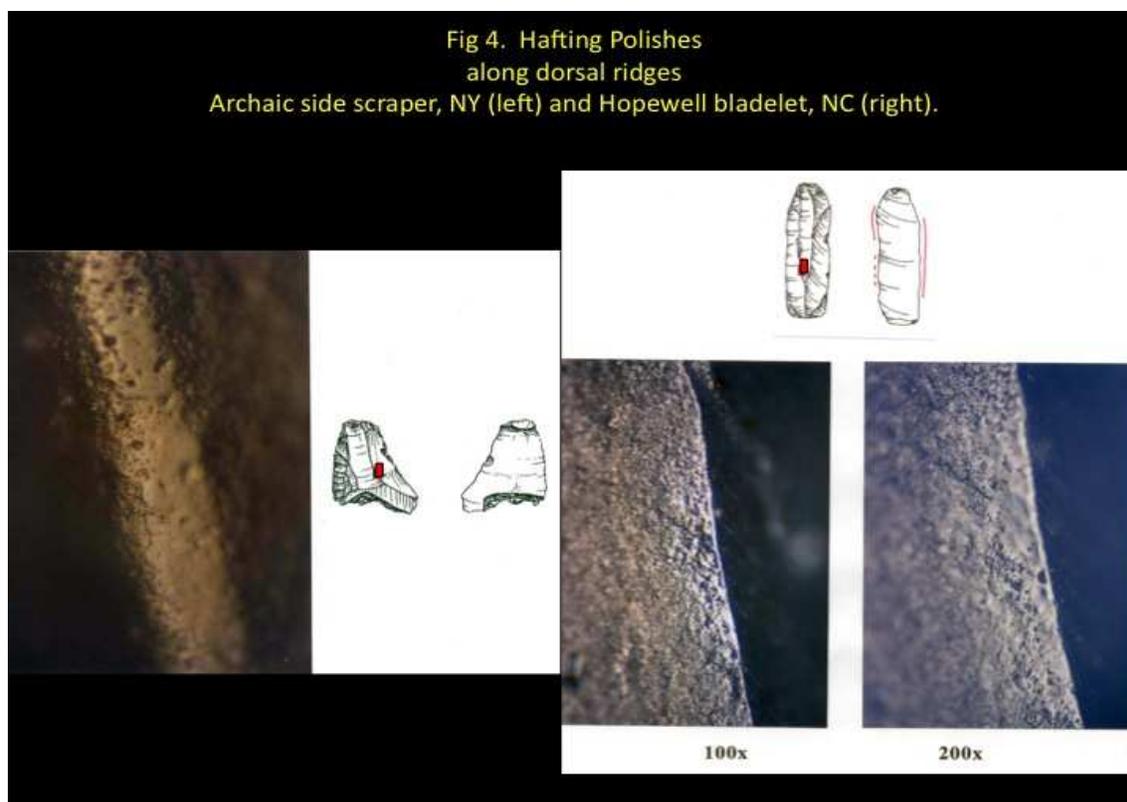


Fig 34. Hafting traces on tools found in America.

In turn, Keeley's results were replicated and extended by a number of microwear analysts in Europe (Anderson 1979; Anderson-Gerfaud 1981; Beyries 1984; Moss 1983; Plisson 1985; Mansur-Francomme 1984; Hurcombe 1986; Juel Jensen 1994; van Gijn 1990) and the United States (Vaughan 1981, 1985; Toth 1982; Yerkes 1987; Donahue 1986; Driskell 1986; Sussman 1986; Kimball 1989; Shea 1991; Sievert 1992). At the same time, Semenov's "low-power approach" (Fig. 33) was replicated by Tringham *et al.* (1974) and further systematized by Odell (1977, 1995) and Shea (1991).

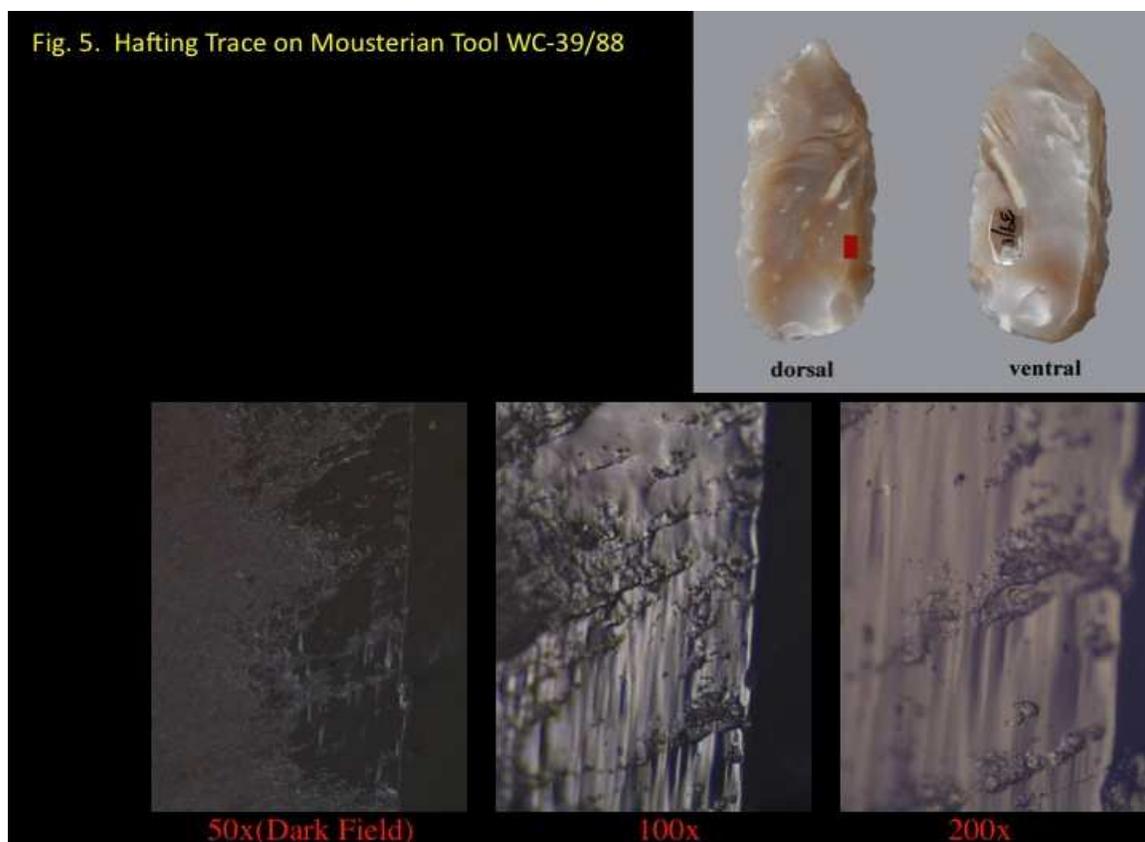


Fig 35. Incident-light microscopy of a hafting trace on Mousterian Tool WC-39/88 (region indicated by red square in upper-right image).

The revision of the Keeley Method used herein is referred to the Keeley-Plisson Method for it combines the experimentally-based system described in Keeley (1977, 1980) and systematically defined by Plisson (1985). Discrete microwear polishes are specifically defined for the attributes of polish *localization*, *extent*, *texture*, *contour*, *brightness*, and *coalescence/polish* (Kimball *et al.* 1995). Kimball (1989) extended the Keeley-Plisson program and added hafting polishes as well (Figs. 34-35), and a systematic, experimentally-based study of hafting traces is now published by Rotts (2010).

Today, the “high-power Keeley Method” is the most generally accepted method for the determination of stone tool function (Juel Jensen 1988; Yerkes and Kardulias 1993). At the same time, analyses of edge damage (Tringham *et al.* 1974), residues (Anderson 1980;

Anderson-Gerfaud 1986; Fullagar 1998; Hardy 1999; Wadley 2005), and chemistry (Evans and Donahue 2005) are parallel approaches, which are increasingly used in tandem with high-power analysis (Anderson *et al.* 1998, 2006; Longo and Skakun 2008; van Gijn 2010; Rots 2010).

Quantitative Approaches to the Description of Microwear Polishes.

The earliest attempts at a quantitative measurement/differentiation of microwear polishes (exclusive of micro-edge damage) seem crude today, but nonetheless highlighted one problem with high-power microwear analysis – that is, the identification of different polishes (i.e., meat, dry hide, fresh hide, wood, bone, antler, soft plant, ivory, etc.) was a qualitative procedure, whose accuracy was admittedly determined by the experience (if not reputation) of the analyst. While Keeley (1977, 1980) was able early on to show quantitative differences in reflectivity of microwear polishes in two clusters (soft plant, antler versus wood, dry hide, and fresh hide) using a photometer and contrasting polish reflectivity for dark-field and light-field reflection in microamperes, most quantitative measurements were made indirectly from microphotographs rather than on the microwear polishes themselves (Dumont 1982a, 1982b; Grace *et al.* 1985, 1987, 1988) or were confined to microflaking (Akoshima 1987). These early attempts at quantifying polishes were largely doomed to move understanding forward because they were based upon image analysis of photographs of polishes rather than direct measurements taken on the polishes themselves.

In addition, very little attention (except for that of Plisson 1985 and Kimball 1989) was placed on an explicit description of the attributes (other than polish) that are, in fact used by the microwear analyst to arrive at a determination of material worked (see above). With

Plisson's (1988a, 1988b) visit to Semenov's traceology lab at Leningrad State University, a new appreciation of the breadth of Semenov's traceological system emerged; and began an integration of both low-power and high-power approaches into a "neo-traceological method." This extended the original traceology of Semenov, while infusing the "post-Keeley approaches" of some late 20th century analysts (Anderson *et al.* 1998; van Gijn 2010; Juel Jensen 1994; Rots 2008). Today, microwear analysis is a more mature science. In addition to the common use of both stereo and incident-light microscopes, the SEM, laser scanning confocal, interferometer, and (as reported herein) atomic force microscopes are being used to study polish characterization and formation – often in quantitative terms. Finally, new ethnoarchaeological (Beyries and Rots 2008) and "reconstructionist" experimental approaches (Skakun 2008) have emerged which further strengthen the overall methodology.

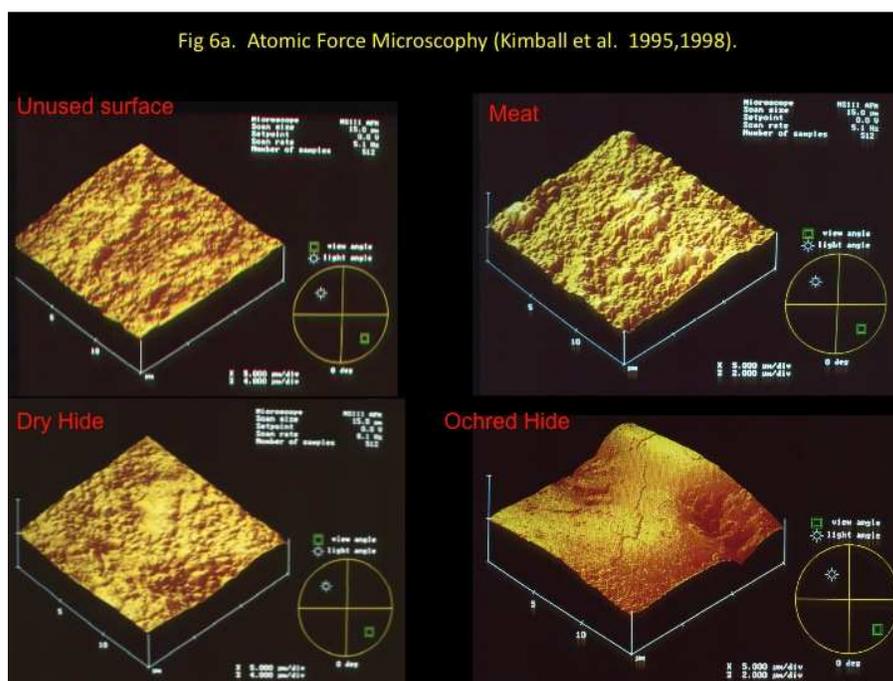


Fig 36a. Ground-breaking quantitative microwear study using AFM to study control regions, meat cutting, dry hide working, and ochred hide working polishes.

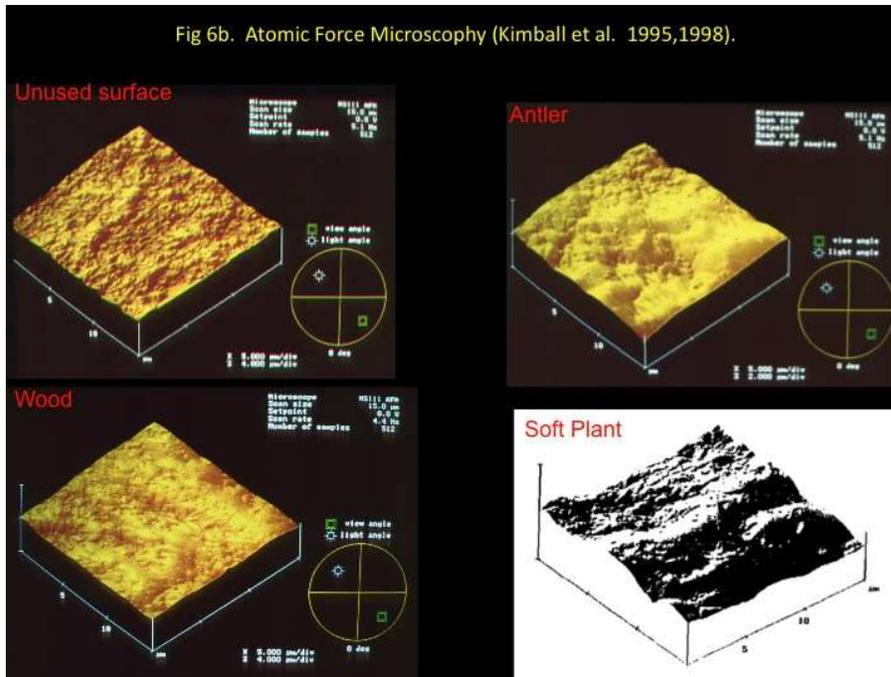


Fig 36b. Quantitative AFM study of control regions, antler polish, wood working, and soft plant polish.

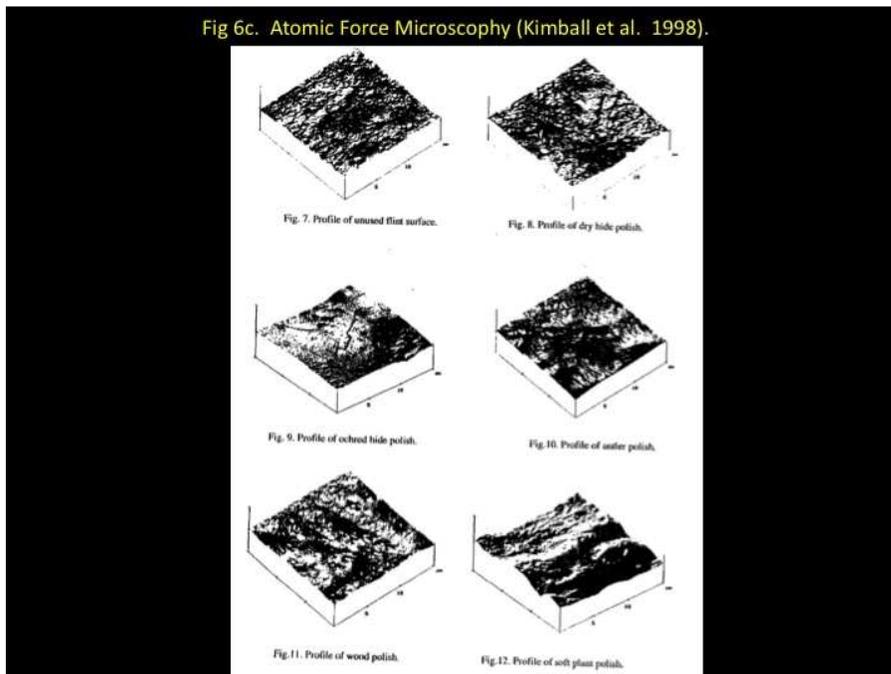


Fig 36c. Quantitative AFM study showing 3d surface maps of various polish regions.

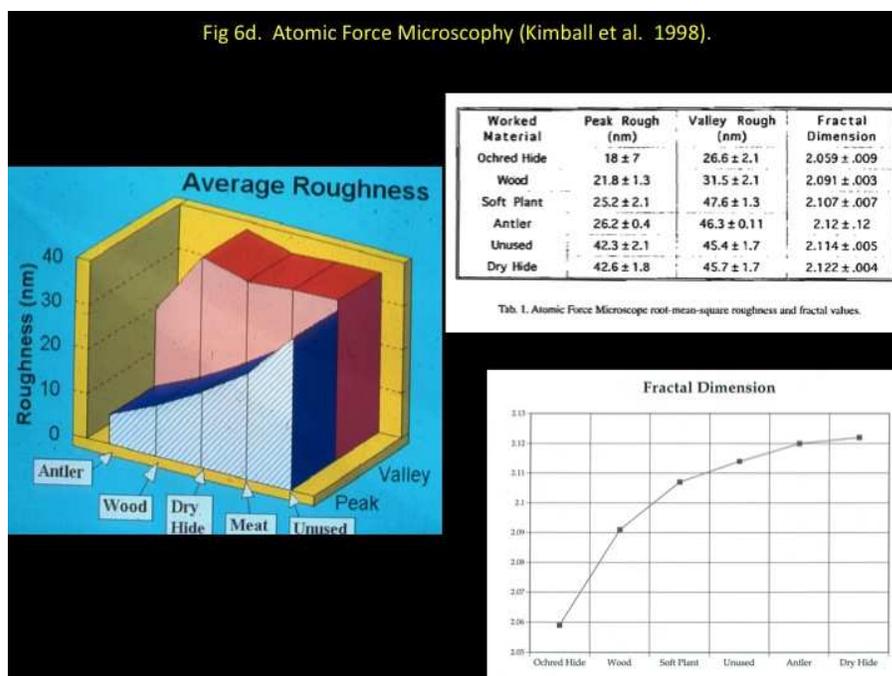


Fig 36d. Results of the first quantitative AFM study of microwear traces on Mousterian tools.

At Appalachian State University, Larry Kimball collaborated with physicist Patricia Allen and her student John Kimball in an unique use of the atomic force microscope (AFM) to scan microwear polishes (meat, antler, wood, dry hide, ochred hide, and soft plant) on experimental tools (Figs. 36a-36d). We felt that the AFM (Digital Instruments NanoScope® III Scanning Probe Microscope) showed great promise in the analytical study of microwear polishes. The results (Kimball *et al.* 1995, 1998) gave some preliminary insight into: (1) how they vary quantitatively; (2) how they can be visually characterized with new attributes; and (3) how microwear polishes formed. The study imaged experimental stone tools with the AFM and from these images determined the average roughness values for flint surfaces' peaks and valleys before and after different types of polishes.

Along with this initial AFM study, a series of very interesting pilot programs began using *optical interferometry* (Anderson *et al.* 1998; Gonzalez-Urquijo and Ibanez-Estevéz 2003), *tribology/vertical-scanning interferometry* (Anderson *et al.* 2006), *laser profilometry*

(Stemp and Stemp 2001, 2003; Stemp *et al.* 2010), and *laser scanning confocal microscopy* (Evans 2008; Evans and Donahue 2008). With the exception of the study by Anderson *et al.* (2006) of a single class of plant threshing tools, none of these programs proceeded to analyze archaeological specimens. And none of these studies produced more accurate quantitative measures of microwear polish differentiation that Kimball *et al.* (1995, 1998).

However, the initial AFM study was constrained by the size restriction of the old stage, which necessitated the use of small (~2 cm) replica tools, and thus prevented the expansion of the findings to actual archaeological specimens. The AFM used in this study is a Veeco Dimension Icon which has a large sample stage that can accommodate tools up to several centimeters in height and up to 20 x 20 cm in size, which allows us to image actual artifacts without cutting them to fit to a small sample holder (Fig. 37).

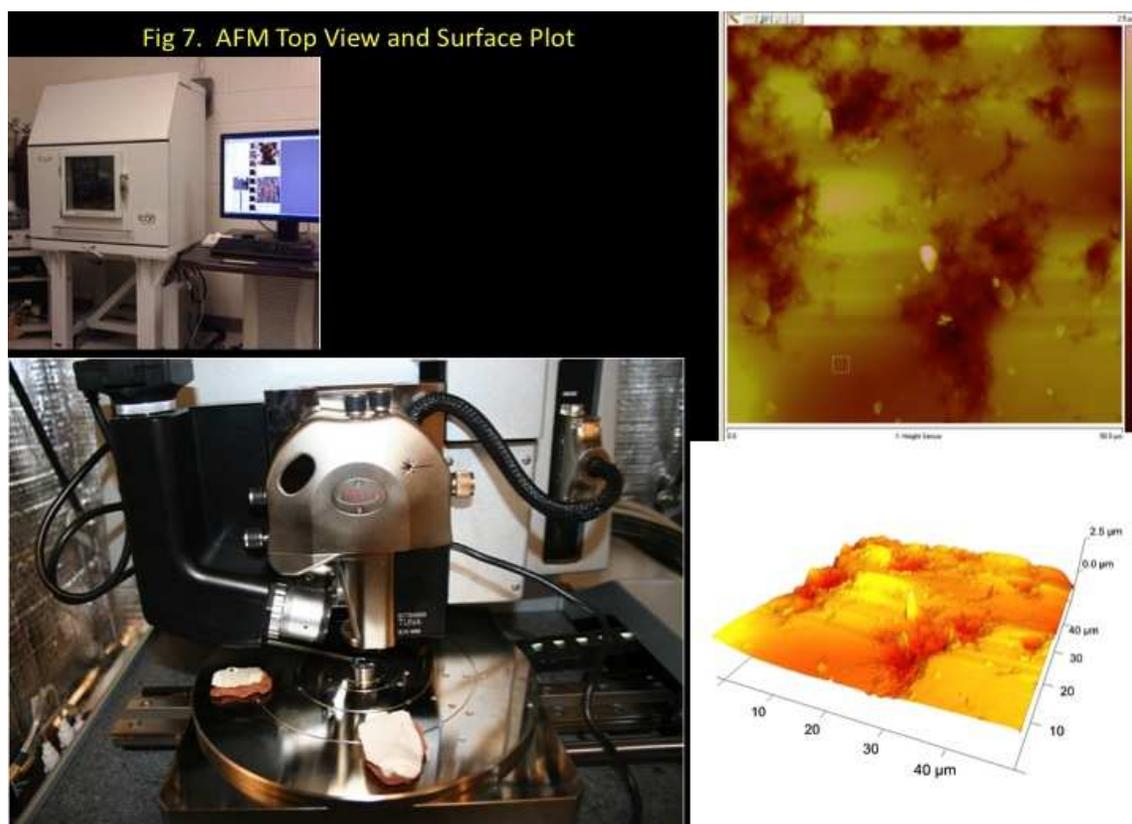


Fig 37. Veeco Dimension Icon AFM and examples of images produced from this study.

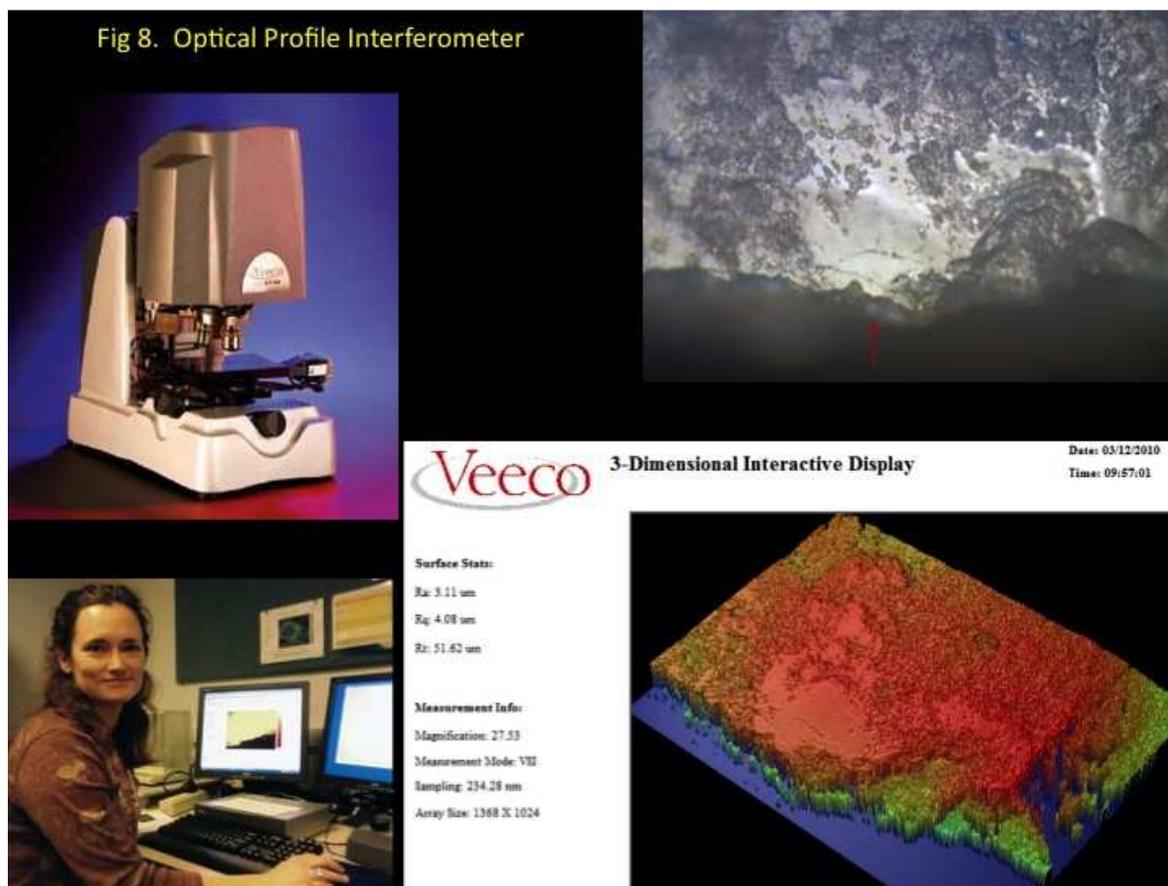


Fig 38. Veeco Wyko Optical Interferometer used in this study, and example images from the interferometer and an incident-light microscope.

The contribution of our new team at Appalachian State University (Tonya Coffey and Nathan Faulks – Physics; and Larry Kimball – Anthropology) specifically focuses on the use of the atomic force microscope, the scanning electron microscope, and the optical profile interferometer (Figure 38) in the measurement of polishes on Mousterian tools (Coffey *et al.* 2010; Faulks *et al.* 2010, 2010a, 2010b, 2011). In this study, we combine the Keeley method with AFM to analyze six different polishes (meat, bone, fresh hide, dry hide, wood, and hafting) on actual archeological artifacts -- the microwear polishes observed to date for the Mousterian assemblage from Weasel Cave.

The Keeley method is ideal for first identifying the locations and discernment of different types of the microwear traces. However, it is based on qualitative-based attributes and does not provide quantitative data and the magnification of typical incident-light binocular microscopes is relatively low. AFM can provide high magnification imaging and quantitative information about the polish topography. However, low magnification images are not always possible as scan head is controlled by a piezo which limits scan sizes to a maximum of roughly 0.1 x 0.1 mm. Also, the piezo limits the heights of the features in the scan that can be imaged to 10 microns or less, depending on the microscope. This means that many use traces cannot be imaged in the AFM, as the tool is simply too rough – thus the AFM appears to be limited to the study of fine-grained flints.

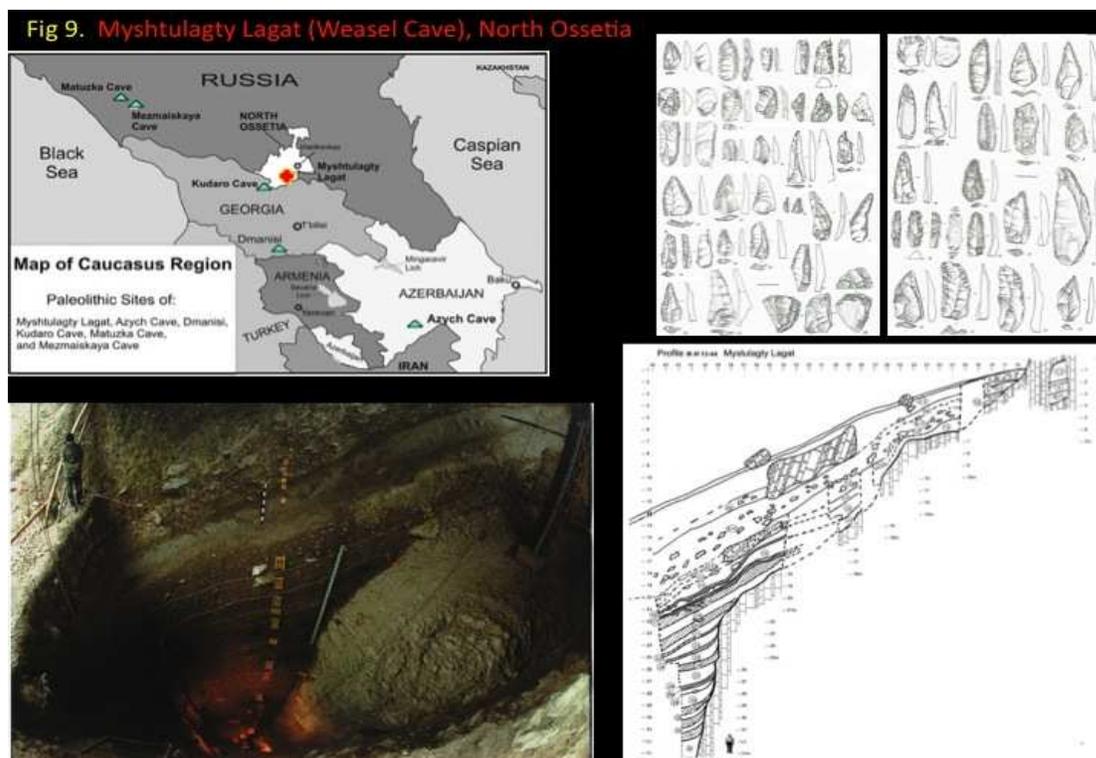


Fig 39. Map of Caucasus region, Russia with profile view of stratigraphy in Myshtulagty Lagat (Weasel Cave), as well as example tools excavated from this region.

Analysis Sample from Myshtulagaty Lagat.

The artifacts studied herein are Mousterian stone tools from Myshtulagty Lagat (Weasel Cave), North Ossetia, Russia (Hidjrati 2003, 2009). The cave is at 1125 m AMSL in the north-central Caucasus Mountains (Figure 39). Preservation is exceptional including carbonized seeds, nuts and wood, and the excellent preservation of microwear polishes on all flint artifacts. Excavations by Nazim Hidjrati since 1981 reveal an intact stratigraphy of over 22 vertical meters representing 36 distinct layers. As of 2010, 23 distinct layers can be assigned to the Middle Paleolithic, and contain Typical Mousterian or Denticulate Mousterian with Levallois blades industries. They span the Middle to Late Pleistocene with Layer 14 dated to Isotope Stage 5e. The tools analyzed in this study represent only six the 20 Mousterian flint tools studied thus far. All 94 flint artifacts, including debitage, have been analyzed by the Keeley Method, which represents 100% of the flint tools excavated to date (Hidjrati and Kimball 2011). A sample of 178 quartzite artifacts, of the thousands recovered, have been analyzed as well. The six Mousterian tools reported herein derive from Layers 12-13, which date to between 50,000 and 90,000 (Isotope Stages 4-5c) based upon pollen and microtine studies. These tools are of Ossetian flint from sources ~20-30 km due west and south of Weasel Cave. It is a very fine-grained and the surfaces appear fresh.

INSTRUMENTATION

Incident-Light Microscope.

After excavation, the Mousterian artifacts from Weasel Cave are cleaned in a weak HCl bath to remove the calcite coating on almost all specimens. The tools were then ultrasonically cleaned in an ammonia detergent bath before inspection for microwear traces

with an Olympus BH incident-light microscope under 50, 100, and 200x. The observed polishes conform to those found on experimental tools by Kimball as well as those expected by a large number of microwear analysts employing this same method of cleaning and microscopy. Each polish is described according to the formal Keeley-Plisson-Kimball schema (Kimball 1989).

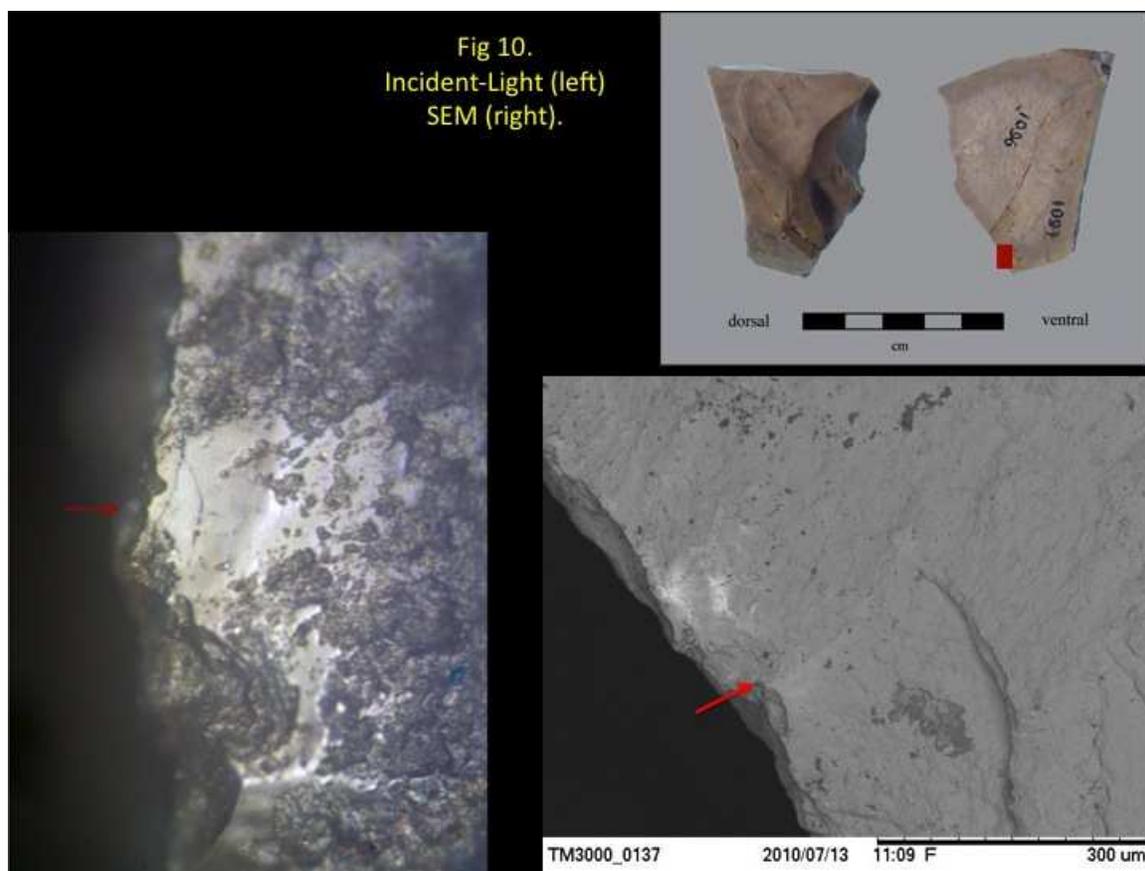


Fig 40. Images obtained from the TM-3000 tabletop scanning electron microscope (wear spot is indicated by red square in upper-right image).

Scanning Electron Microscope.

SEM imaging was performed with a tungsten filament Hitachi Tabletop SEM (TM-3000). The samples were not casted and were not coated, as we did not want to damage or

alter the artifacts. Low voltage (5 kV) was used to reduce sample charging. The SEM images were not filtered or enhanced (Fig. 40).

Optical Profile Interferometer.

A Veeco Wyko Optical Interferometer (at the Tribology Research User Center at the Oak Ridge National Lab) was employed to obtain interferometry for the sample (Fig. 38). The Veeco Interferometer employs coherence scanning interferometry (also known as white-light interferometry) to produce high quality three-dimensional surface plots with sub-nanometer vertical resolution of the scanned tool (Fig. 44). Due to poor optical reflectivity of the Weasel Cave flint tools, we were forced to use data restore on the interferometer images to fill in some missing data points and apply a high band-pass filter to reduce noise. We also flattened the images to correct for the tilt of the sample plane.

Atomic Force Microscope.

Once the polishes were identified via the Keeley method (and scanned using the SEM and interferometer), they were mounted on clay for imaging in the Veeco Dimension Icon AFM (Fig. 37). The Veeco Icon has an optical scope mounted above the sample stage, so that the polishes can be precisely positioned for proper imaging. The samples were imaged in tapping mode. In tapping mode AFM, a tip mounted on a cantilever oscillates at high frequency with constant amplitude. The tip is approached towards the sample until the amplitude of oscillation is damped due to the tip tapping on the sample surface. This constant amplitude of oscillation is maintained by moving the tip up and down in response to surface topography, as the tip is scanned back and forth across the sample surface. The

motion of the tip is controlled by a piezo in a feedback loop with the control electronics. Tapping mode AFM was utilized as opposed to contact mode AFM to ensure that the wear to the AFM tip was minimal; tip wear can limit the accuracy of the roughness measurements. Our AFM tips and cantilevers are commercially available Veeco probes with resonance frequencies of 190 kHz and tip radii of less than 10 nm. The tip was changed frequently to ensure accurate roughness measurements. The AFM images shown have been flattened with a first-order polynomial to correct for the tilt of the sample plane, but have otherwise not been filtered or enhanced. All of the AFM images were acquired at the same image size, 50 x 50 micron, and 512 x 512 pixels, at a scan rate of 0.15 Hz.

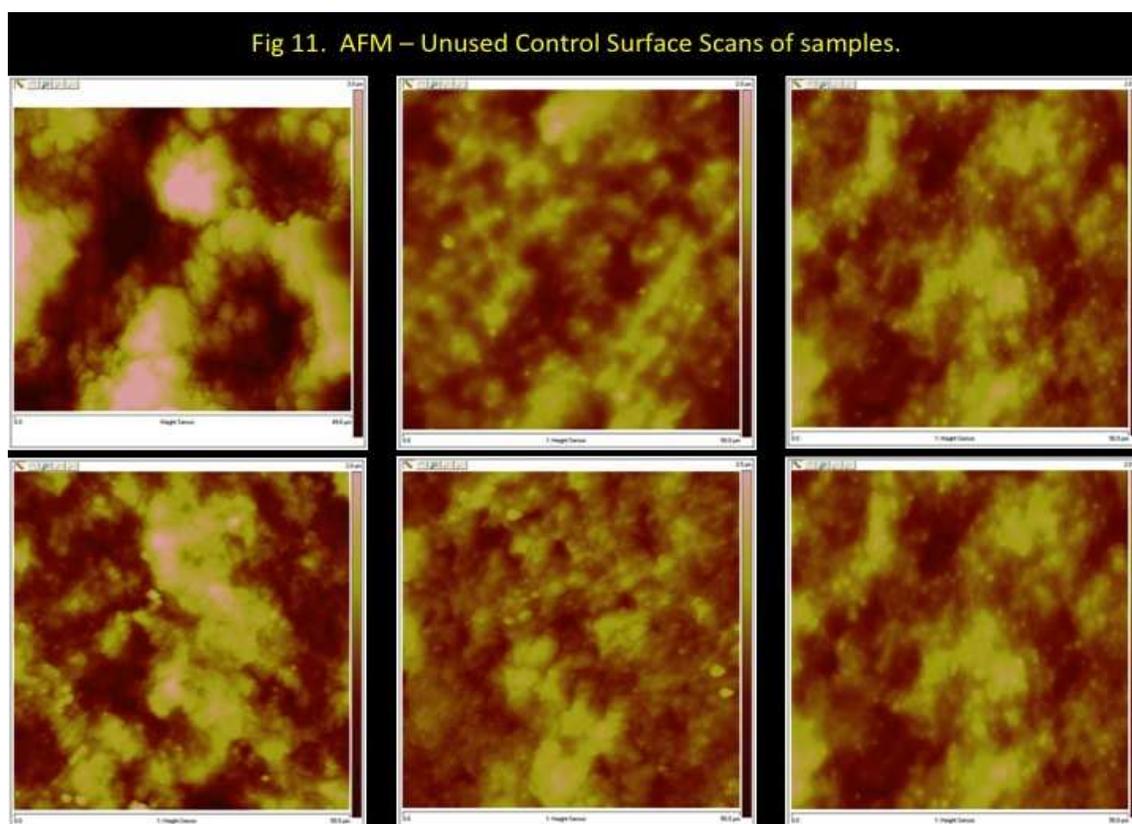


Fig 41. 50 x 50 micron AFM scans of unused control areas on the flint tools. The top-left image was cropped to 45 x 50 micron due to streaking caused by extreme roughness in the microtopography.

The acquired AFM scans were made of both the microwear polishes and unused control areas on the same tool (Fig. 41). The surface microtopography of the polish and unused areas were partitioned into “peaks,” “transitions,” and “valleys” – the highest, sloping, and lowest portions of the surfaces. AFM Nanoscope software was used to calculate the average roughness (Ra) of these peaks, valleys, and transition regions in each scan. Each scan (50 x 50 microns) was sampled by ≥ 10 representative $2 \times 2 \text{ um}^2$ square areas (Fig. 42; small square in bottom right image) of each type to perform this analysis, predicting that the peaks, valleys, and slopes or transitions should be worn at different rates. The results and corresponding uncertainties are displayed in tabular format (Tables III and IV; Figs. 45-50). We report the values of the average roughness, skewness, and excess kurtosis for the 50 x 50 micron AFM scans.

Skewness (Sk) is a measure of the degree of symmetry of the image; images with a Gaussian distribution of surface roughness values have a skewness of zero. Images with plateaus have lower values of skewness than images with isolated steep peaks.

$$Sk = \frac{1}{nR_q^3} \sum_{i=1}^n (Z_i - \bar{Z})^3 \quad (4)$$

Here, n is the number of data points, R_q is the root mean square roughness of the image, Z_i is the height of the i^{th} data point, and \bar{Z} is the average height of the image. Kurtosis (K) shows the pointedness or bluntness of the distribution of the roughness values. Smoother profiles have less variation in their roughness values, and therefore the distribution of roughness values is more narrow than a Gaussian distribution. This causes the kurtosis for more uniform surfaces to be higher than surfaces with greater variation in roughness values.

$$K = \frac{1}{nR_q^4} \sum_{i=1}^n (Z_i - \bar{Z})^4 \quad (5)$$

The resultant AFM scans presented herein are Top Views and Surface Plots. These mediums provide the observer with an intuitive understanding of surface morphology before quantitatively analyzing the surface roughness. Profiles (Section Analyses) are discussed in this paper, but measurements such as Ra, Sk, and K may not be compared or interpreted in the same way for section analyses (see Kimball *et al.* 1995 for examples of surface and section analyses).

RESULTS AND ANALYSIS

Wood-working polish.

Burin WC-1096/1097 is interpreted to have been used to plane wood along three burinated edges, but was not hafted. The scanned Use-Trace 1c is indicated by the red rectangle in the image of the tool (Fig. 42). A digital microphotograph of the microwear polish at 200x is shown in the upper left (the digital image is 344 um in height). The identification of this microwear trace as wood polish is based upon an invasive to spreading *extent*, smooth, united *texture*, a fluid polish with a *coalescence* following the entire microtopography. The *polish* is most pronounced at the higher elevations, and continues over the edge rather than rounding or otherwise significantly modifying it. The *contour* of the polish is irregularly clear. It is a *very bright* polish rarely with striations.

The AFM images were acquired of the polish (Fig. 42) as well as an adjacent unmodified surface representing the control for this tool (Fig. 41, upper left image). [It is important to keep in mind that there is some variation in the graininess of these Mousterian tools as they are on different types of Ossetian flints.] The microwear polish is scanned over a 50 x 50 micron region. The edge of the tool is close to the right location of the area

imaged. The control sample has been cropped to 50 x 45 micron due to streaking in the AFM image caused by the extremely rough surface of the tool. It is easy to see the areas of microwear in these top view scans and 3D surface reconstruction. The peaks of the surface have been almost completely worn away and flattened smooth. The valleys remain rough, with no evidence of in-filling, which is in agreement with the findings of Ollé and Vergés (2008) in their experimental study of polish formation using the SEM. This is reflected in the analysis of the roughness data from the peaks, valleys, and transition regions, as shown in Table III. The average roughness for peaks is dramatically decreased compared to the control image, while the roughness for valleys and transitions is relatively unmodified.

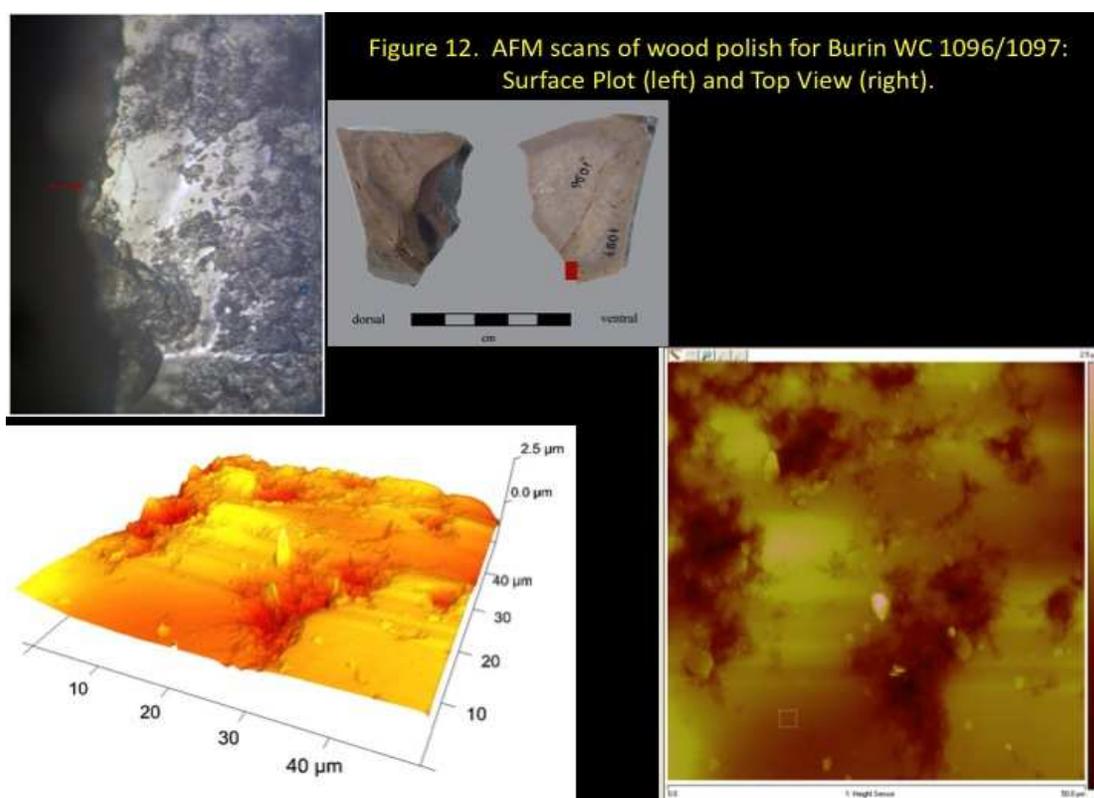


Fig 42. Incident-light and AFM scans of Burin WC 1096/1097, a tool used for wood working.

Figure 44 shows interferometry scans of the wood polish tool. These scans glean different types of information about the surface morphology as they are limited by different surface characteristics. The resolution of the interferometer is typically limited by surfaces with low optical reflectivity, a problem encountered with some polish types. When imaging non-conductive surfaces such as flint tools, the SEM is often limited by charging effects, even in low vacuum and low beam-voltage environments. Brighter regions indicate negative (-) surface charging where more secondary electrons encountered the Everhart-Thornley detector. However, this charging is itself an evidence of wear, because charge bleeds off asperities more readily than from the regions of polish.

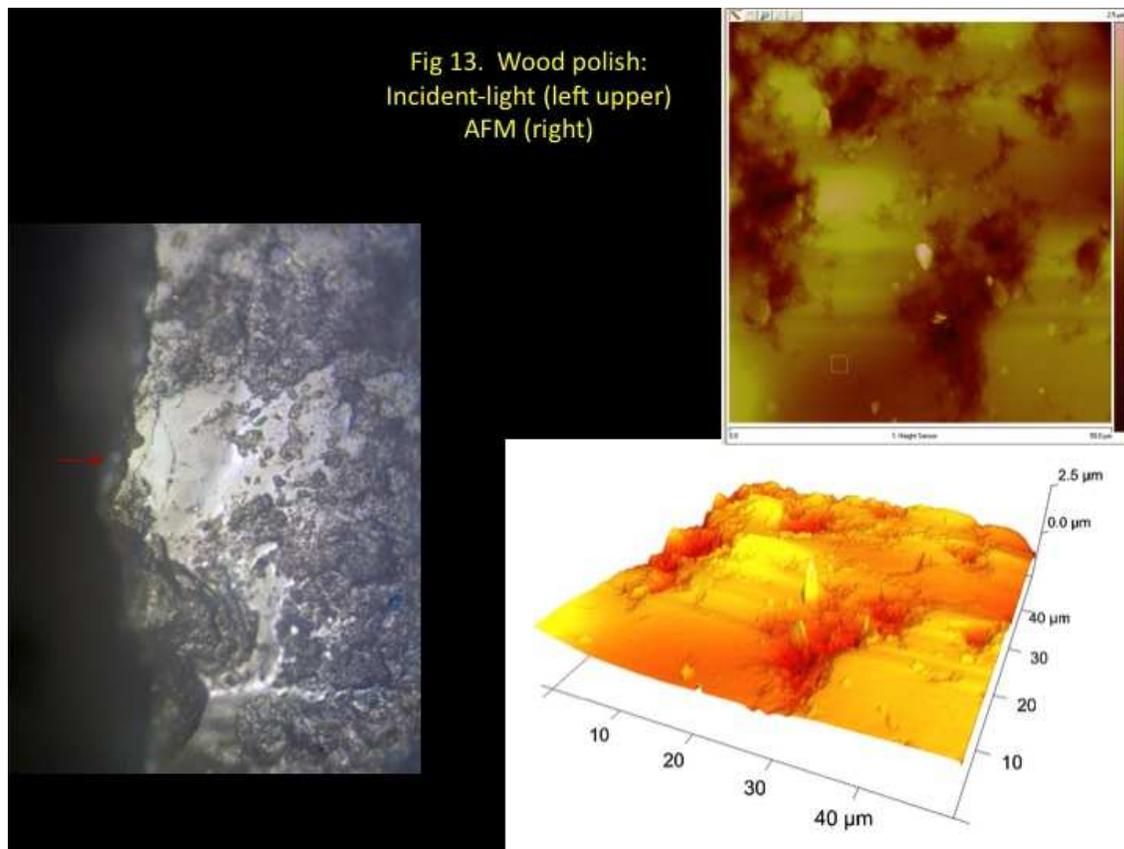


Fig 43. AFM scan of the wood-working trace.

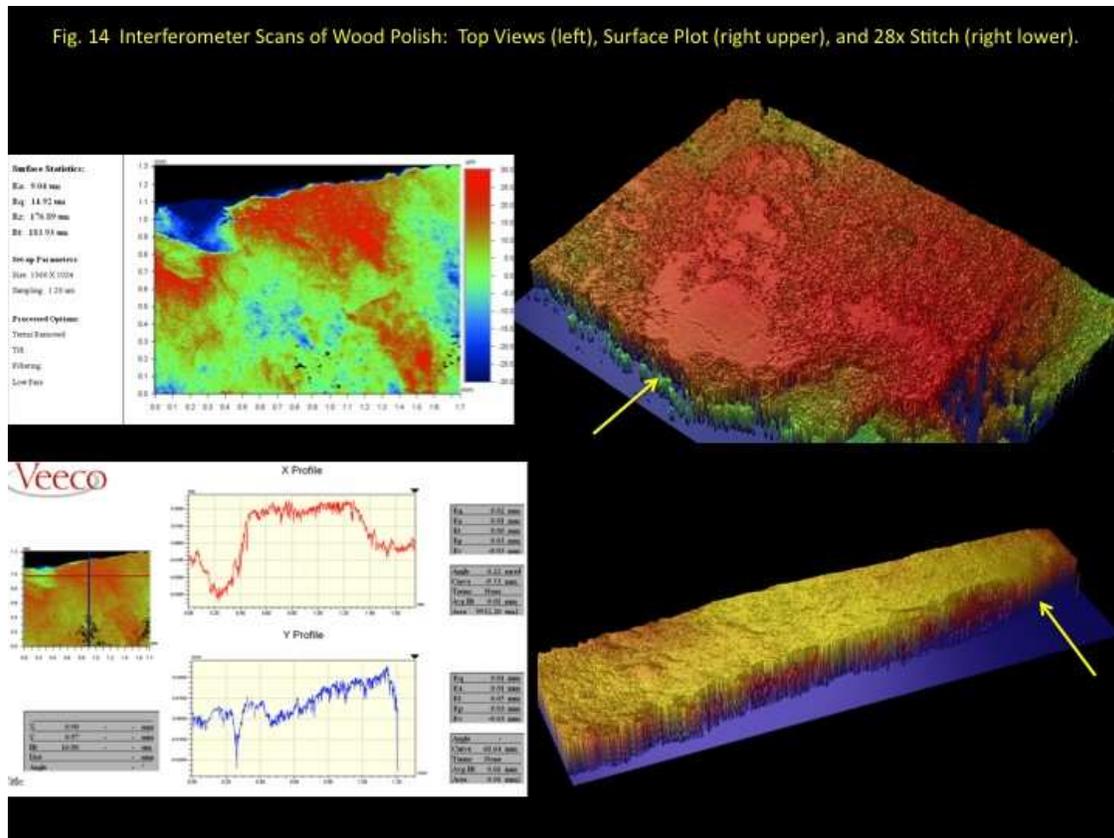


Fig 44. Interferometry scan (upper-right) and multi-scan “stitch” (lower-right) of the wood-working trace.

For comparison, Figures 43-44 show the same polish using the SEM (Fig. 43 lower left) and interferometer (Fig. 44). Note that while quantitative data can be obtained from interferometer scans (but not the SEM), the apparent resolution is not as fine as with the AFM. However, the worn-down aspect of the wood polish is apparent in the interferometer scans (Fig. 44 upper right) and the “stitch” -- multiple scans stitched together over a set distance (Fig. 44 lower right) permits the observation that the portion of the edge where the wood polish has accrued is located at the highest portion of the microtopography along the tool edge.

When we attempted to use SEM to scan the polish region on the wood-working tool, we were limited by a high degree of charging. When imaging non-conductive surfaces such

as flint tools, the SEM is often limited by charging effects, even in low vacuum and low beam-voltage environments. Brighter regions indicate negative (-) surface charging where more secondary electrons encountered the Everhart-Thornley detector. However, this charging is itself an evidence of wear, because charge bleeds off asperities more readily than from the regions of polish.

Fresh hide-working polish.

Atypical Mousterian Point WC-39/1988 (Fig. 45) is interpreted to have been used to clean fresh hide (at the distal end) and whittling wood (along the lateral right edge) in a hafted mode. Use-trace 4b is illustrated here, was used to clean hide in a fresh state – that is, to remove adhering tissues on the interior hide surface. Digital microphotographs of the microwear polish at 200x is also shown (the digital image is 344 um in height). The identification of this microwear trace as fresh hide-working is based upon a fluid and grainy polish following the microtopography of the tool edge. With intensive work, the polish modifies the higher portion of the topography more significantly. Its extent is invasive and exhibits an average texture. The contour of the polish is fuzzy and exhibits an average or “matte” brightness. Striations are present and are short, wide, and deep into the fresh hide polish.

AFM scans were acquired of the microwear polish (Fig. 45) and control (Fig. 41, lower left) over areas of 50 x 50 microns. The edge of the tool is close to the location of the microwear trace is imaged, and is to the right of the image. The AFM image of the microwear trace provides characteristics about the polish morphology that is not easily seen in the optical image. Note the striations and directionality of the wear; the observable

striations within the microwear polish vary from being parallel to at a small angle from the working edge, thus indicating the tool kinematics. This is probably indicative of a cutting motion of the tool through the tissues attached to the hide.

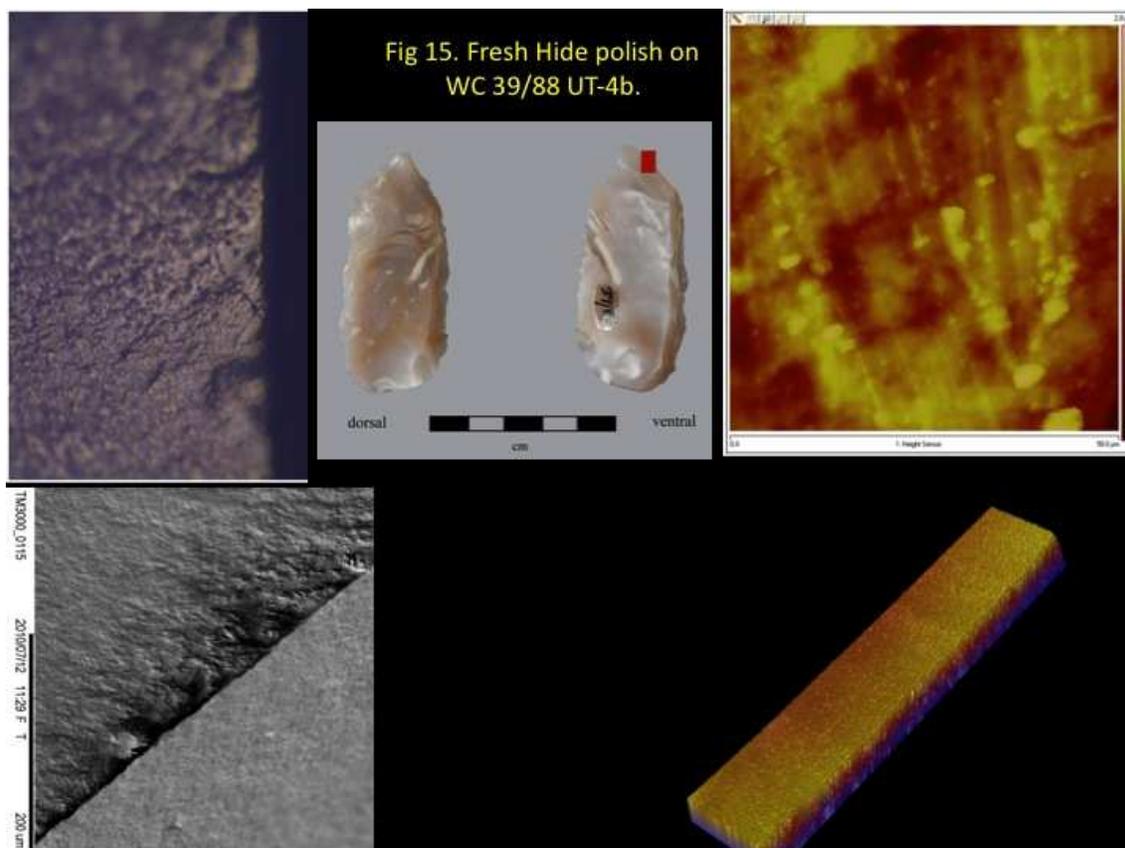


Fig 45. A fresh hide-working tool (WC 39/88) is shown (clockwise from upper right) with AFM, optical interferometry, SEM, and incident-light microscopy.

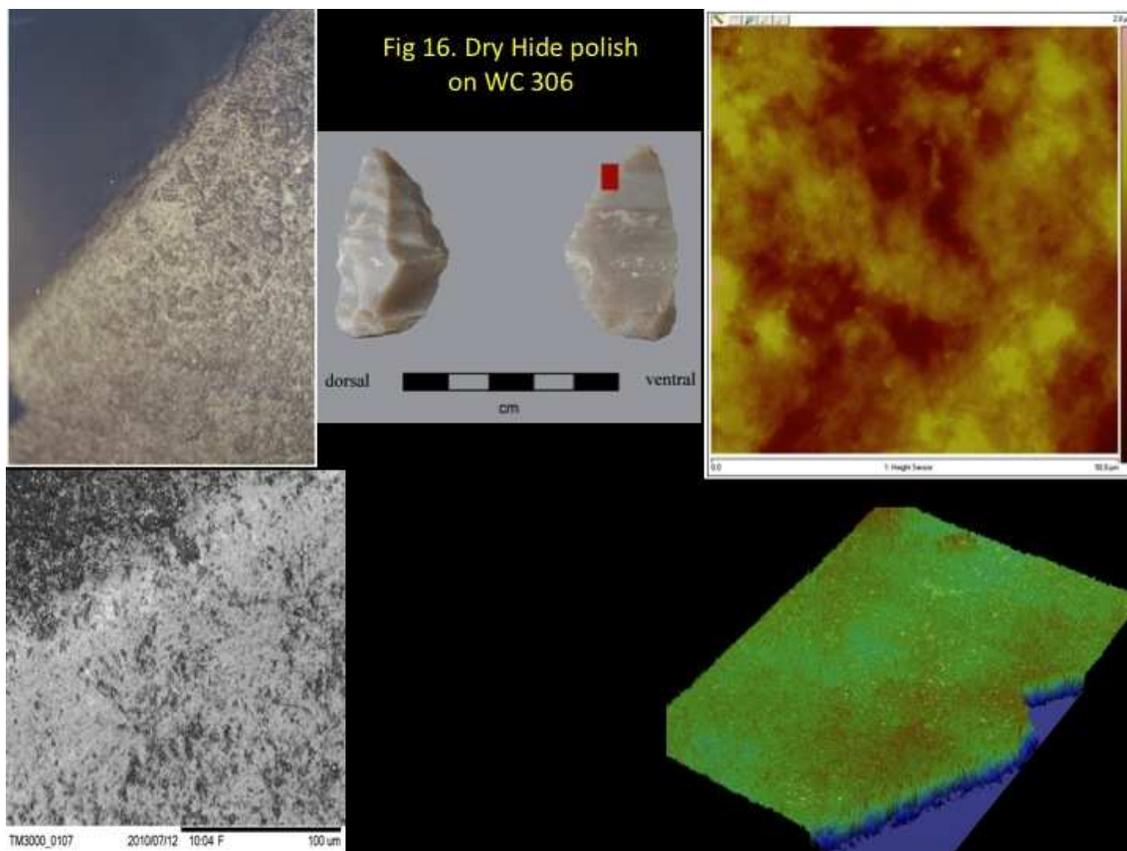


Fig 46. A dry hide-working tool (WC 306/88) is shown (clockwise from upper right) with AFM, optical interferometry, SEM, and incident-light microscopy.

Dry hide-working polish.

Mousterian Point WC-306/1988 (Fig. 46) is interpreted to have been used in two functions: (1) butchery along the lateral right edge; and (2) planing wood along the lateral left edge. The illustrated Use-Trace 3a exhibits dry hide polish in this location (along with microwear polishes from cutting through the hide in fresh condition, cutting through meat, and in one place contacting bone). Digital microphotographs of the microwear polish at 200x is shown in the upper left (the digital image is 344 μm in height). This is the classic manifestation of heavy butchery (Kimball 1989; Yerkes 1987, 1994), as opposed to simply

cutting through meaty tissues. In this case, it appears that processing of the carcass continued until the hide was relatively dry. The illustrated dry hide polish is defined as a soft, grainy polish with significant edge rounding and modification of the microtopography. The texture is dense, the contour is fuzzy, and exhibits a matte/weak brightness. The numerous striations are long, wide, and deep. The extent of the polish is moderate. The image also shows that abrasive wear of the tool edge caused a rounding and smoothing of the working edge of the tool. These observations are clearly evident in both the incident-light and SEM images (Fig. 46).

AFM images were acquired of the microwear polish and an unused control location (Fig. 41, upper middle). Both images are 50 x 50 μm . The edge of the tool to the right of the imaged top view. The AFM scan, and to a lesser extent the interferometer scan, reveals that dry hide working affects all portions of the microtopography, while appearing to make the transitions rougher (Table III).

Meat cutting polish.

Levallois Blade WC-1047/1988 (Fig. 47) is interpreted to have been used in butchery (lateral right edge) and wood planing (proximal edge). Use-trace 6b is identified as a polish from cutting meat, but all along this working edge meat and fresh hide polishes were observed. The identification of this microwear trace as meat is defined by a very fluid polish which affects the entire microtopography alike without major alteration of the relief. The texture is dense and the contour is fuzzy, but more evident at 50-100x. Yet, it forms a continuous linear band of polish along the working edge. The brightness is average and

somewhat lustrous. A few striations are evident and they are narrow, straight, and short; and indicate the direction of tool use.

AFM images were acquired of the micropolish (Fig. 47) and an unused control location (Fig. 41, lower right), and are 50 x 50 μm . The edge of the tool is to the right of the image. The AFM image of the polish gives information about its morphology that is not so easily seen in the optical image – the striations which indicate the directionality in the polish. There are two distinct striae, running both parallel (top of image) and oblique (bottom of image) to the edge. It seems as though the tool was used in a cutting motion, which would give striations parallel to the edge, and then later more in a slicing motion, giving oblique striations relative to the edge.

The average roughness values for this example of meat polish show a smoothing of the peaks and valleys, indicating that the entire microtopography is worn. The wear is not as dramatic as in some of the more abrasive polishes, but it is measureable within standard deviations. Although many microwear analysts are reluctant to identify microwear traces from processing meat, it can be measured.

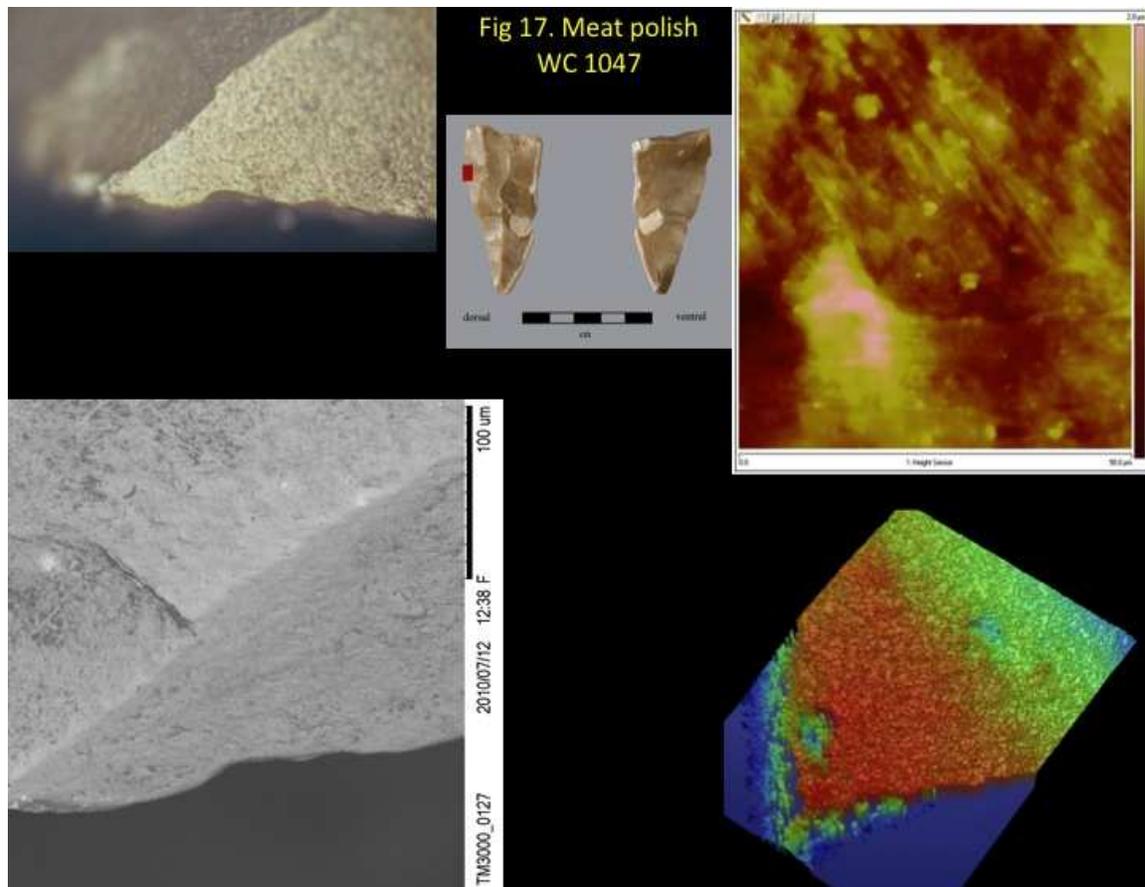


Fig 47. A meat cutting tool (WC 1047/88) is shown (clockwise from upper right) with AFM, optical interferometry, SEM, and incident-light microscopy.

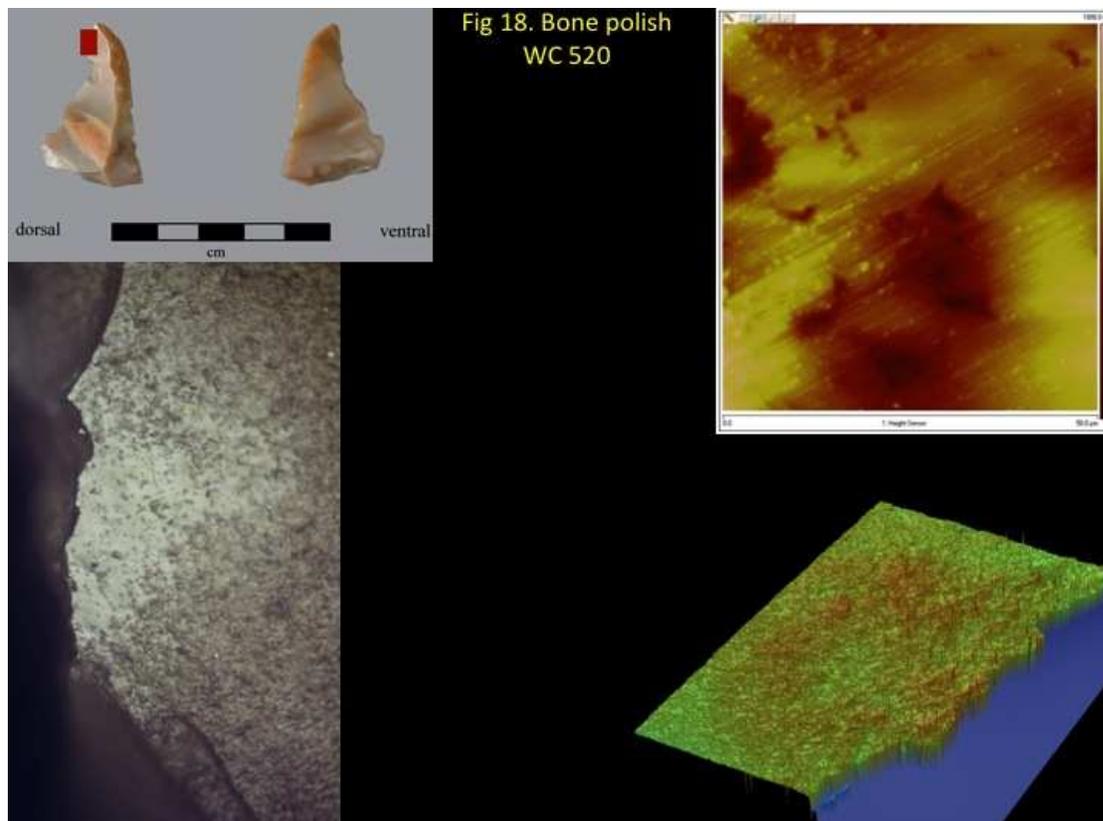


Fig 48. A bone polish tool (WC 520) is shown (clockwise from upper right) with AFM, optical interferometry, and incident-light microscopy.

Bone polish.

Atypical Levallois Point WC-520 (Fig. 48) was observed to have been used in butchery (both lateral edges) in a hafted mode. This is indicated by the presence of fresh hide and meat polishes continuously distributed along the working edge, and the occasional spots of bone polish (see Kimball 1989, 1994; Yerkes 1987, 1994). At the illustrated location a spot of bone polish was observed at the distal lateral left point. Elsewhere along both lateral edges, polishes from cutting through fresh hide and meat were observed. At an isolated location, bone polish is a hard, undulating and bright polish that spreads across the microtopography. It also exhibits the classic attributes of “pitting” (Keeley 1980). Bone

polish concentrates at high elevations of the microtopography and at projections along the edge. The contour is clear and rather abrupt. Bone polish is observed experimentally to have an isolated extent in sawing/cutting actions. The texture is dense.

AFM images of the bone polish region (Fig. 48) and the unused control region (Fig. 41, lower right) were acquired for 50 x 50 um areas. The edge of the tool is to the left of the scan. Bone polish is similar to the wood polish in that the microwear is much more abrasive, resulting in a polish that is very smooth and flat. However, this example of bone polish has a distinct directionality to small striations, oriented oblique relative to the edge. This indicates that the tool was used in a slicing motion into the bone, as one would expect during butchery.

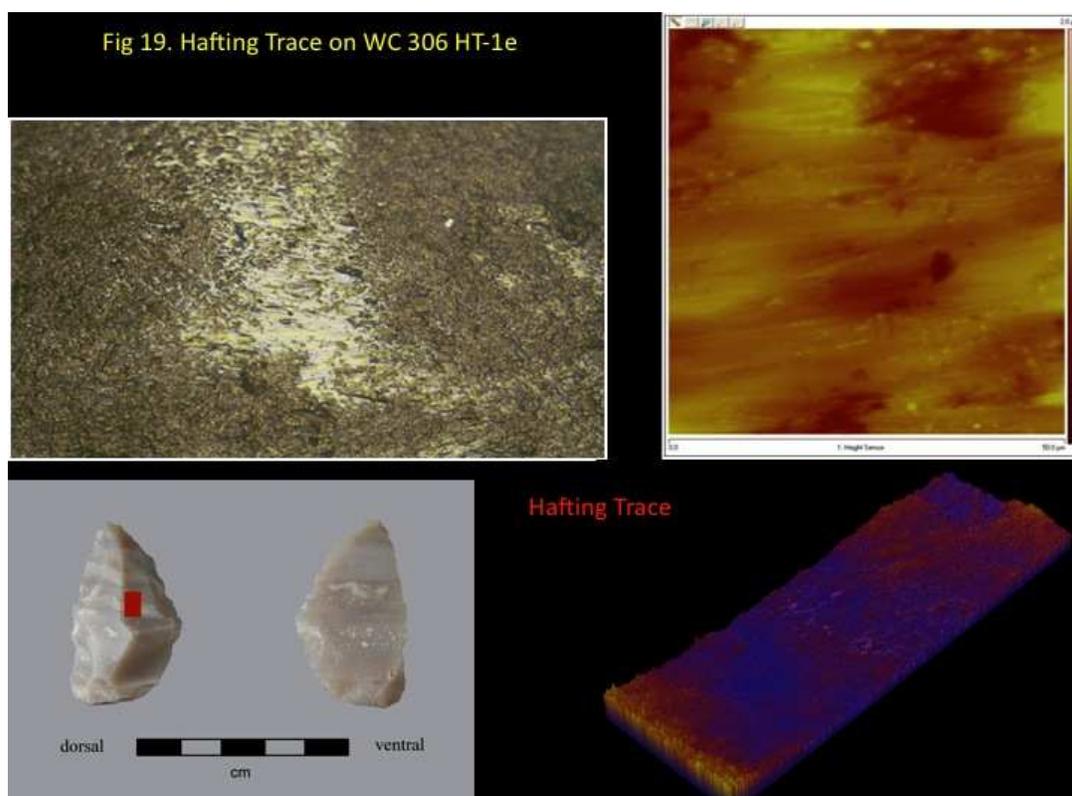


Fig 49. A tool with a hafting trace (WC 306/88) is shown (clockwise from upper right) with AFM, optical interferometry, and incident-light microscopy.

Hafting polish.

Some stone tools, such as Mousterian Point WC-306/1988 (Fig. 49), contain hafting traces at Weasel Cave (79 %), evidence of wear created by the positioning of the tool in a wood or bone handle. They are evidenced by a very smooth, brilliant polish, striations, and modifications of the tool edge by micro-scarring or edge-rounding (Rots 2008, 2010). When viewed at the correct angle a hafting trace can be highly reflective (Fig. 50). Dominant variables in the formation of hafting traces are the hafting material, the hafting arrangement, fine included materials (dust, sediment, ochre, organic materials, etc.), and the nature, force, and duration of work. The coarseness and morphology of the tool prior to use are secondary variables in determining the characteristics of the polish. The optical image shows Hafting Trace-1e on the prominent dorsal ridge at 100x magnification. The microtrace is very reflective and exhibits directionality of the polish.

AFM images of the hafting trace (Fig. 49) and the control (Fig. 41, upper middle) are presented. Both images are 50 micron x 50 micron and have z-scale bars on the right side. While the control image is topographically unaltered, the hafting trace shows visible wear scars with directionality. This is significant in identifying the type of wear spot because the edge of the tool is just above the top of the image. The wear scars are parallel to the edge of the tool, a common evidence for characterizing hafting traces.

A second, and dramatic (yet not rare), example of an archaeological hafting trace is observed on the Mousterian Point WC 39 (Fig. 50) with incident-light and dramatic interferometer images presented. The advantage of the interferometer is clear here as a very large portion of the hafting trace can be considered and, in our opinion represents a very

heavy wearing down of the high portions of the micro-relief. The directionality of the undulations of this hafting polish are parallel to the axis of work.

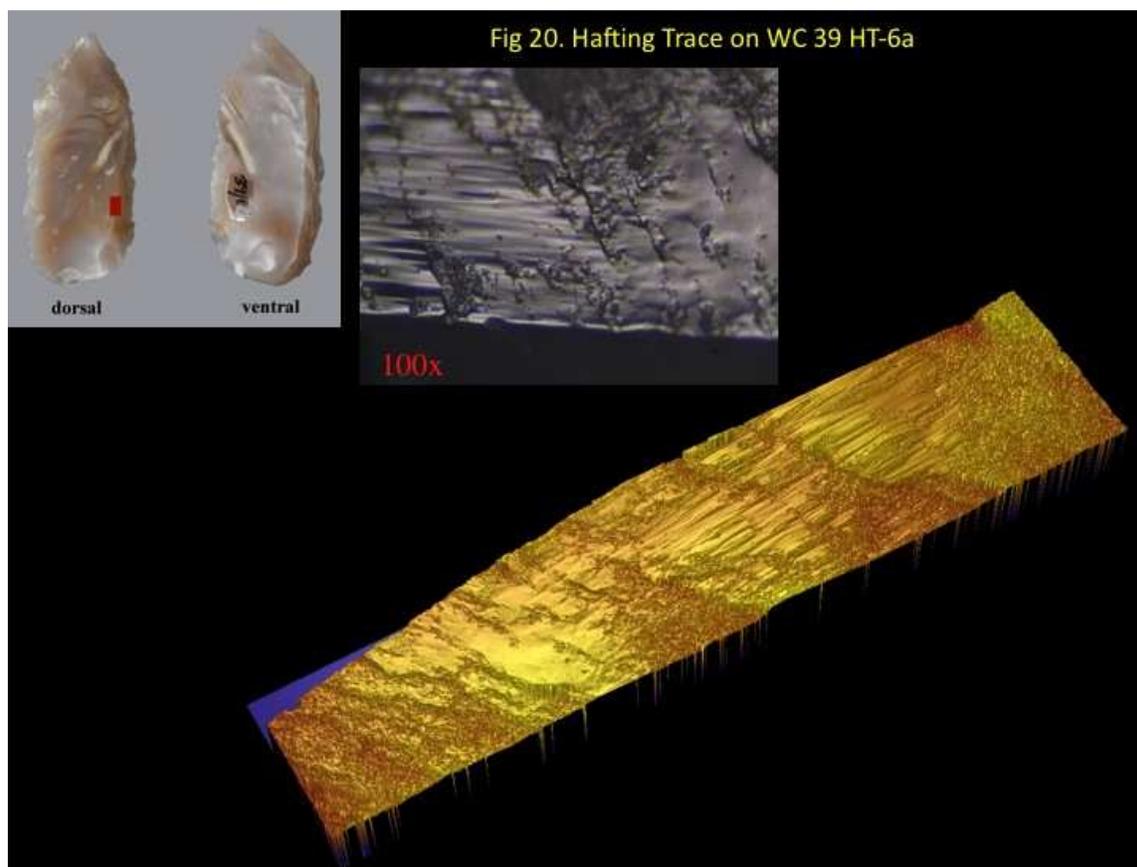


Fig 50. A tool with a hafting trace (WC 39/88) is shown with incident-light microscopy (upper) and optical interferometry (lower).

QUANTITATIVE ANALYSIS

The previous AFM study on experimental flint tools (Kimball *et al.* 1995, 1998) showed that the surface roughness was lowest for tools used in working antler, followed closely by wood working, then dry hide working, and finally meat cutting was the least developed polish. Our AFM study of Neanderthal stone tools closely agrees with this. The roughness of the peaks as determined by AFM for the bone working and wood working

polishes are the same within the uncertainties, followed by fresh hide, dry hide and then meat. In the Mansur-Francomme (1983) study on experimental stone tools, it was noted that when there is moisture present during use the wear is more extreme and less localized to the peaks. This agrees with our results for differentiating microwear polishes from working animal hides in fresh and dry states. Studying the morphology from the images of the various techniques, it can be seen that the wear is more uniform and widespread for the fresh hide working than for the dry hide working.

For all of the microwear traces, the skewness determined from the AFM images is reduced for the polish vs. control portions (Figs. 51-56). Since the skewness is lower for surfaces with more plateaus and higher for surfaces with isolated steep peaks, lower values of skewness imply a more worn surface. For all of the traces, the excess kurtosis determined from the AFM images is increased for the polish vs. control regions. An increase in kurtosis implies a more uniform surface roughness. Skewness and kurtosis are therefore parameters that can help give a quantitative measure to distinguish use traces for both experimental tools and archeological artifacts. It is exciting that the less obvious polishes, such as meat or dry hide, show quantitative differences in these parameters, as these polishes are difficult to verify qualitatively by simply viewing the morphology or examining the roughness alone.

Stemp and Stemp (2003) conducted a study with experimental stone tools used to work wood and sherds, and used profilometry to make quantitative measurements of the surface topography. They characterized the degree of wear of the surface using the fractal dimension, and showed that the fractal dimension increased with increasing use of the tool for sawing pottery and for sawing wood. However, they did explain that for the more subtle wood polish as opposed to the more extreme pottery polish, that the changes were not

significant based on the uncertainties in the experiment. In our study with real artifacts, we report similar findings. For the more subtle polishes, the meat and dry hide, the fractal dimension actually decreased for the wear spot as opposed to the control spot. For the wood polish, the fractal dimension did increase, and the differences between the fractal dimensions for the wear vs. control are large enough that they are most likely significant. However, for the bone and fresh hide polishes, although the fractal dimension increased, the differences for the fractal dimension for wear vs. control spots are not as large. Given the inconsistency and variability in the results, we do not recommend the fractal dimension as a good quantitative measure of wear for archeological artifacts.

The data obtained from the Optical Profile Interferometer show many similar trends compared to the AFM topography analysis. Average roughness is decreased, skewness is decreased, and excess kurtosis is increased for the polish vs. control regions on all types of wear. The interferometry results (Table V) seem measurable and consistent within experimental uncertainties, and we recommend this method as a successful quantitative measure of wear for flint tools.

Table III. Average roughness measured by AFM for peak, valley, and transition zones.

Polish Type	R_a (nm) Peaks	R_a (nm) Valleys	R_a (nm) Transitions
Wood Working	8 ± 2	113 ± 9	40 ± 5
Wood Control	52 ± 7	70 ± 9	56 ± 6
Fresh Hide Working	19 ± 3	33 ± 2	22 ± 3
Fresh Hide Control	38 ± 5	54 ± 8	45 ± 5
Dry Hide Working	24 ± 3	31 ± 3	25 ± 3
Dry Hide Control	27 ± 4	26 ± 3	26 ± 3
Meat Cutting	33 ± 3	38 ± 6	29 ± 5
Meat Control	41 ± 4	51 ± 6	26 ± 4
Bone Working	12 ± 2	23 ± 3	12 ± 2
Bone Control	23 ± 3	28 ± 3	25 ± 3
Hafting Trace	18 ± 4	21 ± 3	11 ± 2
Hafting Control	27 ± 4	26 ± 4	26 ± 3

Table IV. Roughness (R_a), Skewness (Sk) and Kurtosis (K) for AFM samples.

Polish type	R_a (nm)	Sk	K
Wood Working	200	-0.027	0.69
Wood Control	622	0.093	0.49
Fresh Hide Working	169	-0.361	0.28
Fresh Hide Control	274	0.192	-0.52
Dry Hide Working	185	-0.340	0.10
Dry Hide Control	180	0.008	0.03
Meat Cutting	214	0.227	1.78
Meat Control	157	0.446	0.08
Bone Working	110	-0.438	0.30
Bone Control	189	0.142	-0.10
Hafting Trace	105	-0.324	0.72
Hafting Control	180	0.008	0.03

Table V. Roughness (R_q), Skewness (Sk), and Kurtosis (K) for the optical interferometry line scans.

Polish type	R_q (microns)	Sk	K
Wood Working	0.36	-1.07	1.16
Wood Control	1.70	0.02	-1.02
Fresh Hide Working	0.45	-0.59	1.31
Fresh Hide Control	0.70	-0.09	-0.37
Meat Cutting	0.50	-0.30	0.36
Meat Control	0.55	0.01	-0.53
Bone Working	0.51	0.02	0.47
Bone Control	0.78	-0.15	-0.39

CONCLUSIONS

The AFM and interferometer provide interpretable, quantitative information in the study of microwear polishes of archeological artifacts at the very small scale. As opposed to other micro- and nanoscale techniques, such as scanning electron microscopy, it can provide quantitative roughness analysis without sputter coating the artifact or casting. The AFM can also provide higher resolution images than optical interferometry or laser scanning confocal microscopy, techniques commonly used for quantitative analysis of lithic artifacts.

Several preliminary conclusions are possible from this on-going analysis of Mousterian tools:

(1) Virtually all microwear traces analyzed to date (including meat polish) exhibit quantifiable differences.

(2) Certain characteristics of these polishes are observable when using the AFM, interferometer, and the SEM – thus complimenting those commonly observed with light microscopes.

(3) While it is premature to discuss the merits of competing models of microwear polish formation (i.e., Anderson *et al.* 2006 versus Ollé and Vergès 2008), our evidence thus far suggests that polishes formed from working hide, wood, bone, and meat appear to be wear. Much systematic experimentation with multiple instruments is needed to better ascertain the nature of this important issue.

(4) There are clear advantages to multi-instrument observation, and each provides important data to not only diagnose what material was worked by stone tools, the kinematics, intensity and duration of use, whether a haft was employed; but also idiosyncratic details that

may well provide insights into ancient stone tool use beyond those expected by current archaeological and ethnographic models.

We are currently pursuing more studies with experimental stone tools made from the types of flints used by these Neanderthals, in order to better compare known stone tool function and duration of use for experimental tools vs. artifacts. This is necessary because it has been shown that the polish development can vary greatly depending on the properties of the lithic raw material.

The different techniques used to analyze stone tools such as AFM, optical interferometry, laser scanning confocal microscopy, SEM, and incident-light microscopy, have their own advantages and disadvantages, and different types of information that can be obtained using each technique. We therefore believe it is important to use multiple instrumentation to develop a truly comprehensive quantitative microwear method.

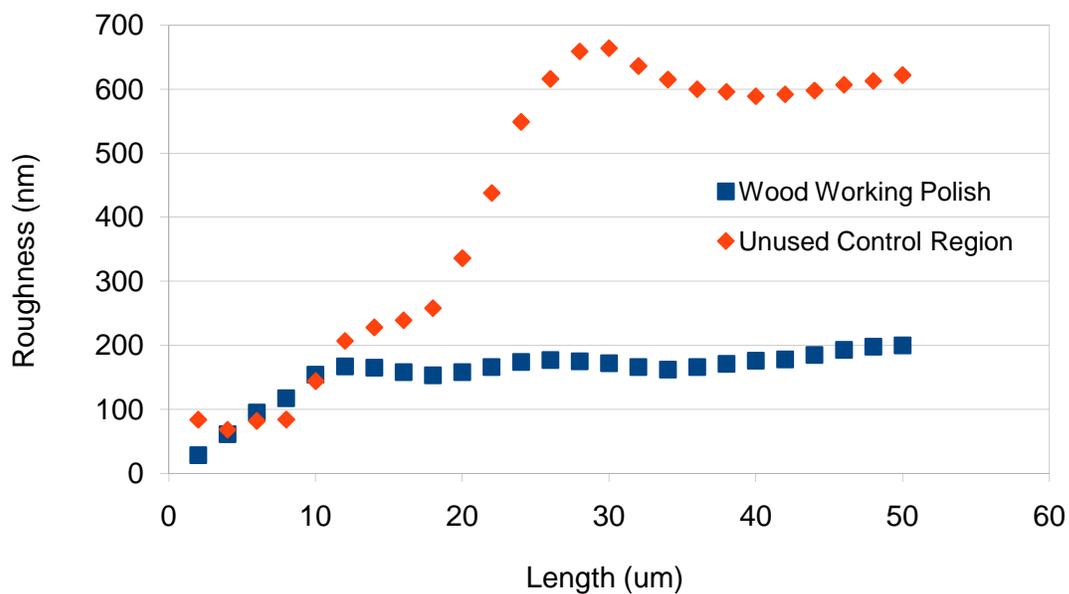


Fig 51. Average roughness as a function of length-scale for a wood-working tool.

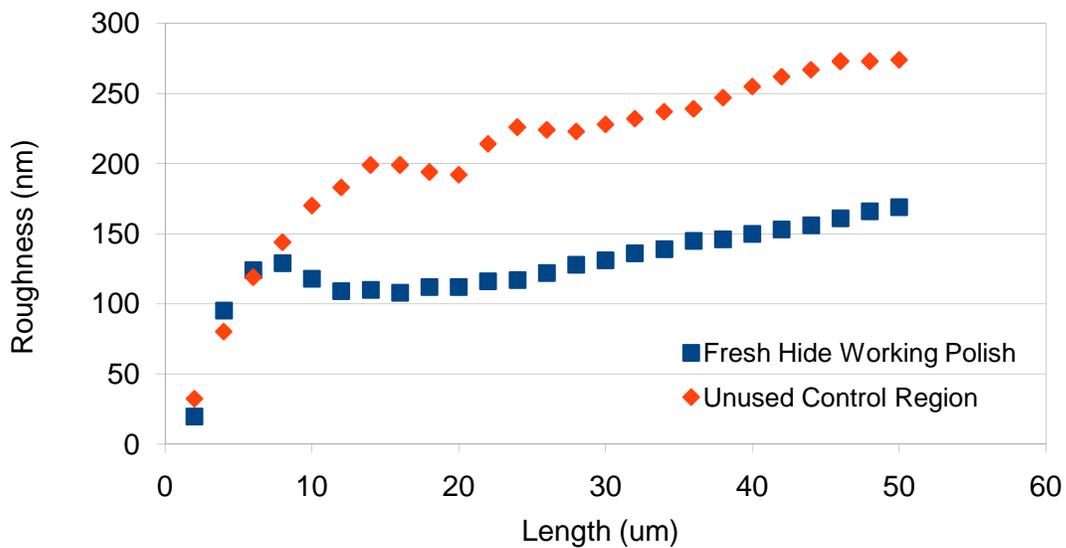


Fig 52. Average roughness as a function of length-scale for a fresh hide-working tool.

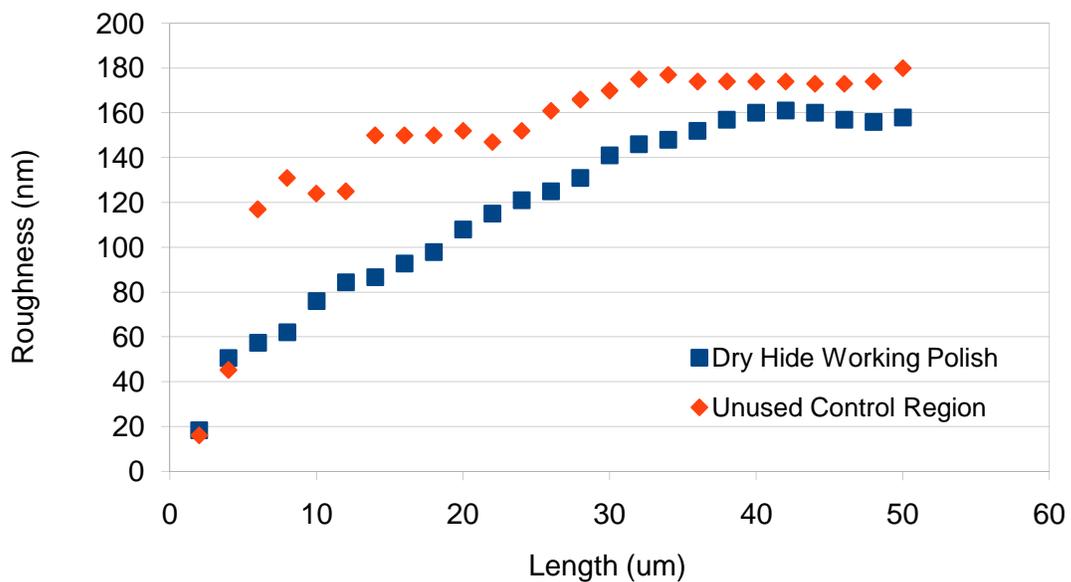


Fig 53. Average roughness as a function of length-scale for a dry hide-working tool.

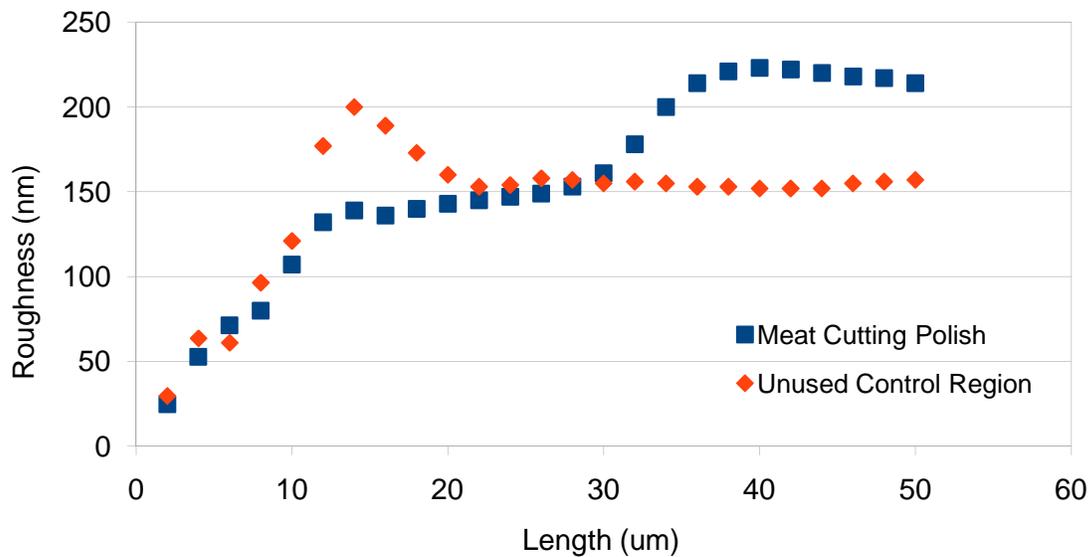


Fig 54. Average roughness as a function of length-scale for a meat cutting tool.

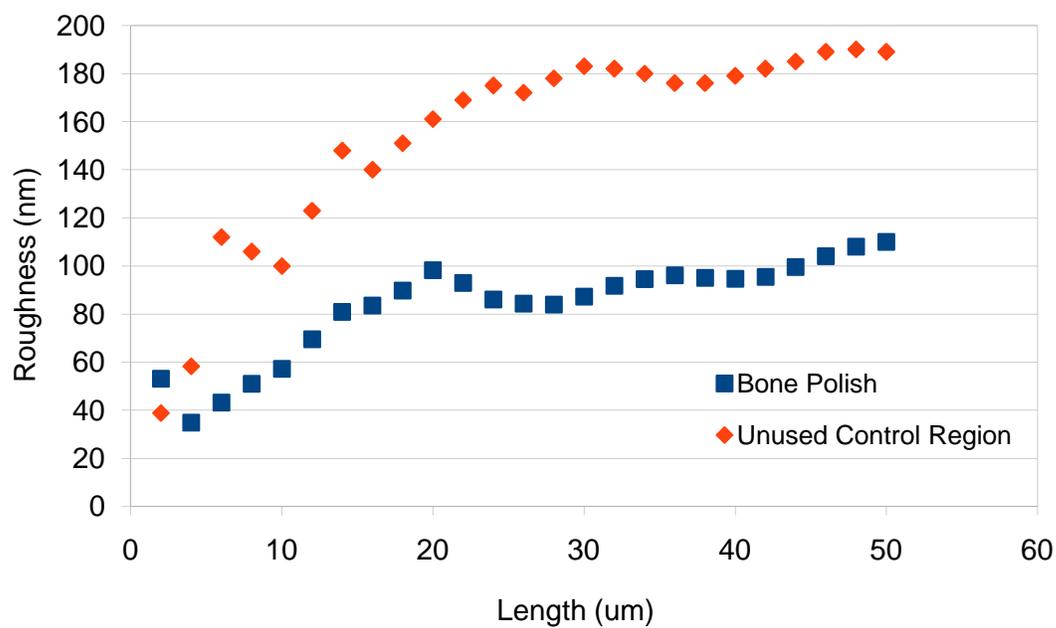


Fig 55. Average roughness as a function of length-scale for a bone polish tool.

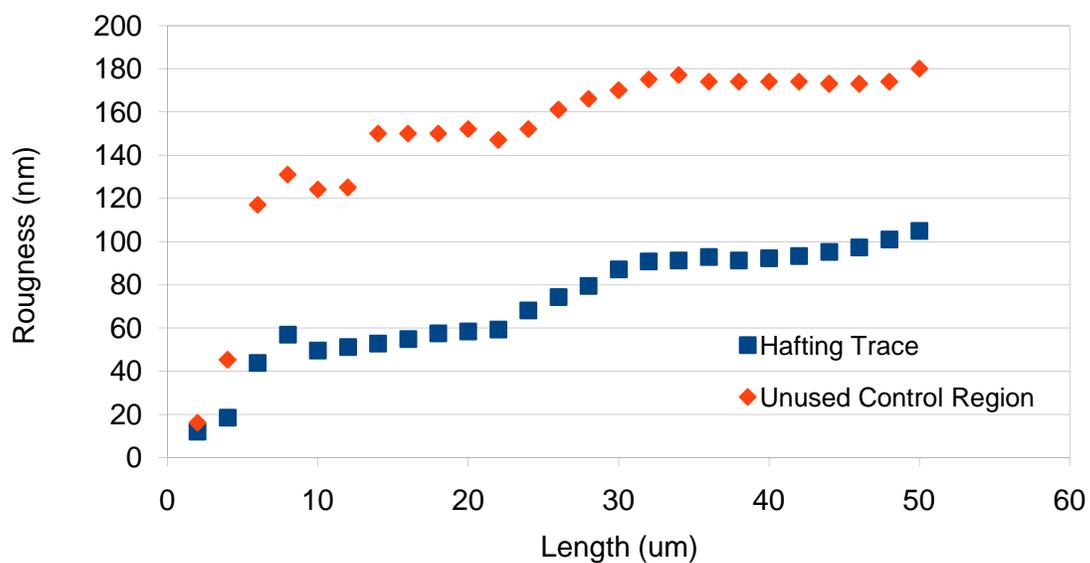


Fig 56. Average roughness as a function of length-scale for a tool with a hafting trace.

CHAPTER 5: CONCLUSIONS AND FUTURE WORK

This thesis used a Veeco Icon Atomic Force Microscope (AFM) to educate K-12 and undergraduate students about the nanoscale world (chapter 2) and to perform archaeological research (chapters 3 and 4). These applications align well with the goals of the National Nanotechnology Initiative (NNI), which coordinates and promotes broad applications and educational strategies for nanotechnology in the USA.

The educational resource developed in chapter 2 was titled “Nanotechnology Learning Modules.” It served as a creative method to provide hands-on nanotechnology education for undergraduate students. Most people have neither the education nor the access to a state-of-the-art AFM system. This learning module attempts to provide just that – an experimental approach to learning about AFM and its many applications. This is a great opportunity because it allows students to connect mathematical and physical principles with a real-world application. The module was field-tested in an upper-level undergraduate course (Experimental Methods in Physics) in the Physics and Astronomy department at Appalachian State University (ASU). Most of the students in the Experimental Methods class had never studied or worked with SPM technology previously, so their feedback helped to enlighten the developers about areas needing clarification. Future work should include the development of an additional section in the learning module on AFM control parameters. Students should be better informed about how variables such as gain, scan speed, and drive amplitude will affect image acquisition.

Future work is certainly needed to develop effective SPM educational resources and to implement new strategies training the next generation of technicians, engineers, and scientists. The number of hands-on SPM educational opportunities are few, and the funding

needed to augment these facilities is limited. Thus, it seems that a cost-effective strategy for implementing SPM education is the continued development of hands-on learning modules using currently available SPM instrumentation and professionals. A critical next step for students studying nanotechnology at ASU is the addition of more hands-on interaction for our students and in outreach activities with K-12 students and the general public with available microscopes, such as AFM, STM, SEM, and FIB.

The research summarized in chapters 3 and 4 used AFM as one of several techniques for classifying the use of Neanderthal flint tools. These stone tools, extracted from Weasel Cave in Russia, were identified as being used for tasks such as meat cutting, dry/fresh hide scraping, wood planing, and others. Depending on the type of flint and the task involved, various degrees of abrasion occurred, leaving behind microwear polishes. These microwear traces are localized regions where the degree of polish is strongly influenced by the task being performed. In the past, most flint tool-use classification schemes were qualitative: a trusted expert performed a visual categorization using a stereo-light microscope. However, the potential for subjectivity in this method called for a repeatable, quantitative analysis technique. The research presented in this thesis attempts to advance the study of microwear analysis using both qualitative and quantitative techniques: incident light microscopy, AFM, SEM, and optical interferometry. Using statistical analysis of roughness, skewness, and kurtosis, measurable differences are shown between tools identified as being used for different tasks. This is exciting because it indicates the success of quantitative microwear analysis in determining flint tool use.

The successful differentiation of microwear polishes can be expanded and verified by comparing polishes on experimentally produced tools with those on excavated artifacts. If

possible, future work should be performed on experimental and actual tools simultaneously to reduce variance in methodology. Quantitative comparison to experimental tools on similar types of flint is an important step, especially when analyzing very subtle polishes such as meat cutting and dry hide-working. This is because it increases confidence in the qualitative analysis performed by an expert. Future work on the stone tools excavated from Weasel Cave should also include the development of a larger database of quantitative data for multiple polish types, using AFM, optical interferometry, and other proven quantitative techniques such as laser scanning confocal microscopy. Due to the large degree of variance in the lithic raw material, polish development can vary greatly depending on the type of flint used. To compensate for this uncontrolled variable, a larger set of data is needed to verify the trends observed in these inaugural studies.

In 25 years AFM has grown from a Stanford University lab to use around the world in government, industry, and academia. AFM provides high resolution topographical mapping at the micro and nanoscale. It extends the reaches of scanning probe microscopy to liquid, ambient, biological, and nonconductive environments. And as demonstrated in this thesis, AFM can even be used to map the very rough surface morphology of flint tools. AFM is an advisable technique for many applications, and it is likely to serve on the frontlines of high-resolution microscopy for years to come.

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VITA

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Mr. Faulks is an active member of Alliance Bible Fellowship in Boone, NC. His parents are Mr. William R Faulks and Mrs. Ellen S Faulks of Nashville, TN.