Journal of Archaeological Science 34 (2007) 711–722
http://www.elsevier.com/locate/jas

Lithic raw material physical properties and use-wear accrual

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Received 3 January 2006; received in revised form 29 June 2006; accepted 29 July 2006

Abstract

Understanding the nature of the physical properties of lithic raw materials is a pre-requisite for developing more reliable interpretations of use-wear evidence and tool function. We use nanoindentation and use-wear experimentation as a way to measure differences in raw material surface hardness and roughness in order to show that differences in lithic material properties have implications for rates of use-wear accrual.

Keywords: Raw material; Hardness; Roughness; Nanoindentation; Use-wear

1. Introduction

Archaeologists have searched for analytical methods that will yield increasingly detailed and accurate inferences about the prehistoric past. There has been considerable innovation in use-wear analysis over the last 50 years (e.g. Grace, 1989; Kimball et al., 1995; Stemp and Stemp, 2001, 2003; Evans and Donahue, 2005). Reliable interpretations of use-wear on stone tools rest on a solid understanding of the physical properties of the raw materials used in tool manufacture. Without an appreciation of how the nature of a given lithic material influences the development of use-related wear, any inferences about tool function should be considered preliminary at best.

In many archaeological contexts lithic tools are the most abundant (and sometimes the only) source of information about prehistoric technological systems. A highly categorical approach has traditionally been used when extracting data from the products of lithic technology: lithic materials are divided into types that are loosely defined in either geological (Quartz, Chert) or functional terms (for example the ubiquitous "polished slate" of Northern Europe); tools are divided into types despite great morphological variability from region to region and within individual sites; use-wear is divided into types according to visible polishes and striations.

Below we explore a less categorical, more continuous and more explicitly quantitative approach to assessing the physical variability of lithic raw materials both within and between types. Given the internal heterogeneity of lithic raw material sources and the idiosyncrasies of the technological process (including tool production and use), an individual stone tool can be seen as a unique combination of raw material structure, composition and morphology. Each stone tool has a unique life history.

Since even the highest quality lithic raw materials are still heterogeneous, it is important to get an idea of the variability of raw materials both in their composition and in their structure. Because even the most rote and mechanical task can be performed in a number of individually unique ways, it is important to consider the very high variability of use-wear outcomes that result from encounters between structure, composition, morphology, and use in a particular social and economic context.

Micro-indentation represents one technique that can lead the way to more continuous and better-quantified analyses of the complex relationship between stone tools and social

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0305-4403/$ - see front matter
doi:10.1016/j.jas.2006.07.009

systems. By measuring differences in properties such as surface hardness and roughness, greater insight can be gained into the nature of use-wear accrual. This increased insight will better equip researchers to evaluate the full significance of use-wear evidence. In the present investigation four different lithic raw materials were subjected to micro-indentation hardness testing in order to gauge variability within and between materials and its potential implications for assessing patterns of wear accrual. Experimental flake tools made of those materials were then used on dry ungulate hide in order to gauge differences in wear accrual and relate them to raw material properties.

2. Literature

Use-wear analysis has continued to grow as a distinct subdiscipline of both lithic and experimental archaeological research. Early debates over which visible aspects of wear are the most reliable indicators of tool use behaviour (e.g. Tringham et al., 1974; Odell, 1979; Odell and Odell-Vereecken, 1980; Keeley, 1980; Vaughan, 1985) have now been largely resolved through widespread acknowledgement that combinations of characteristics are more informative than any individual variable (e.g. Kay, 1996; Jahren et al., 1997; Dubreuil, 2004; Rots, 2005). Shea (1992) recognized the importance of assessing all traces of wear but underscored the need for caution in making functional assessments given the problem of equifinality in the archaeological record. He suggested, however, that as analytical procedures and our understanding of wear formation processes improve; use-wear analysis would become an increasingly valuable interpretative tool (Shea, 1992, p. 150).

The last two decades have seen a shift in research effort towards both greater systematization and diversification in the way use-wear is evaluated. This growth has taken many forms including the development of several original techniques for identifying and distinguishing between different wear patterns and the increasingly precise quantification of these differences. Both of these aspects are equally vital to the overall development of use-wear analysis as viable and productive field of archaeological research.

Keeley (1980) broached the subject of more rigorously quantifying observed differences in wear formation as part of his landmark study of wear patterning as a function of contact material. He cites the use of a light meter to measure polish reflected light levels in the microscopic field of view as a potential method for further differentiating wear patterns in conjunction with a series of more qualitative indicators (Keeley, 1980, pp. 62–63). Keeley, however, did not pursue the issue much beyond stressing the need for more rigorous measures. Since then, use-wear methodology has garnered some additional attention in the work of Dumont (1982), Grace et al. (1983, 1987), and Grace (1989, 1990).

As predicted by Shea (1992), the progress of modern technology continues to foster the development of increasingly sophisticated forms of use-wear analysis. Building on previous work dealing with the nature of polish formation (e.g. Del Bene, 1979; Diamond, 1979; Kamminga, 1979). Fullagar (1991) conducted experiments using a scanning electron microscope to examine the role silica plays in this process. Considering both organic and non-organic sources of silica, he found that a complex relationship exists between this mineral and how a polish develops. One obvious implication is that more developed polishes are better indicators of worked material (Fullagar, 1991, p. 21), but even then they are not entirely free of ambiguity.

Kimball et al. (1995) examined experimentally generated polishes using an atomic force microscope, paying particular attention to differences in surface roughness. Several researchers had previously used scanning electron microscopes to study use-wear traces (e.g. Keeley and Newcomer, 1977; Meeks et al., 1982; Mansur-Franchomme, 1983; Unger-Hamilton, 1984; Knutsson, 1988; Fullagar, 1991), but Kimball et al. (1995) point out that despite an improvement in resolution offered by SEM analysis it is still largely qualitative in nature (Kimball et al., 1995, p. 9). They developed a means of quantifying different use-polishes by measuring and plotting the roughness of their respective microtopographies. They found noticeable differences between polishes produced by working different contact materials, but their technique has yet to see further testing on archaeological material.

Derndarsky and Ocklind's (2001) work on subsurface use-damage in quartz tools moves beyond experimentation to include a full archaeological case study. Using confocal microscopy they were able to generate "stacks of optical sections taken at successive focal planes [that] can be processed to produce a three dimensional view of the specimen" (Derndarsky and Ocklind, 2001, p. 1150, brackets added). They essentially scanned each tool at various depths of field and combined them to form a 3D view of any wear patterns. Using replicated tools they experimented with sawing, cutting, threshing and hide preparation in order to assess the nature and extent of variability in subsurface wear formation.

After comparing the experimental tools with an assemblage of quartz scrapers from Gorviksudden, Tasjo Parish, in northern Sweden, they found that wear associated with different tasks could be distinguished based on the presence, size, number of subsurface cracks and the appearance of pits and disturbed surface areas (Derndarsky and Ocklind, 2001, p. 1157). The implications of their results are, however, presently unclear due to the lack of quantification of subsurface wear traces and by complication that will likely arise when applying their approach to more opaque lithic raw materials such as certain varieties of chert or flint.

Stemp and Stemp (2001, 2003) approached quantifying tool surface roughness through UBM Laser Profilometry. Despite some complications due to certain surface irregularities, they were able to quantitatively assess microtopographic characteristics produced as a result of sawing sand-tempered pottery, conch shell and soaked deer antler (Stemp and Stemp, 2001, 2003, pp. 85, 86). They also carried out additional research examining the process of use-related micro-polish development on an English chalk flint (Stemp and Stemp, 2003). Both Kimball et al. (1995) and Stemp and Stemp (2001, 2003) have proposed intriguing new approaches to use-wear analysis that aim to provide more rigorously quantitative assessments of wear development and variability.

Evans and Donahue (2005) examined patterns of wear based on the elemental chemistry of traces. They found that even following extensive cleansing, organic residue could still adhere to tool surfaces and thus affect wear appearance. This, they suggest, is of critical importance in any attempt to more precisely quantify wear traces in order to maximize their interpretive reliability. All of the studies just discussed share the goal of maximizing the amount of cultural information derivable from a fragmentary archaeological record. Innovative methodologies and the means to implement them in as rigorous manner as possible are essential if we are to truly understand the behaviours represented by the tools we study.

One aspect of wear accrual none of these studies address directly is the role of lithic raw material properties in use-wear formation and development. There has been experimentation in the past dealing directly with how the physical properties of raw materials may affect the generation, and appearance of wear traces (e.g. Goodman, 1944; Greiser and Sheets, 1979; McDevitt, 1994), but these investigations have been few and far between.

3. Theory and Experimental Design

The use-wear on prehistoric stone tools can be used to study human behaviour as represented in the archaeological record. To facilitate this, the physical properties of stone tool raw material must be investigated in order to fully understand the development of wear during use. Hence, the mechanical properties of the tool itself are of great interest, especially its surface properties (e.g. hardness, smoothness).

Indentation techniques are widely used in tests of the surface properties of materials. Starting in the late 19th century, the currently accepted experimental technique began to be developed and has since been refined to produce a very precise method of measurement. Following such experimentation, their theoretical underpinnings were also formulated along the same lines as the elastic/plastic theories in mechanics (Johnson, 1987).

It is obvious that inference of the nature and duration of prehistoric tool use depends on many physical properties, with Young's modulus E and hardness H_c being two of the most important factors. Values for both of these, along with other measures, can be obtained from indentation tests. In solid mechanics, Young's modulus is a measure of the stiffness of a given material and is defined:

$$E = \frac{F \cdot L}{A \cdot \Delta L} \tag{1}$$

where L is the equilibrium length of a sample, ΔL is the length change under the applied stress, F is the force applied, and A is the area over which the force is applied. Young's modulus is measured in units of pressure. A higher value for Young's modulus indicates stiffer and harder materials. On the other hand, hardness is not an intrinsic material property dictated by precise definitions in terms of fundamental units of mass, length and time. A hardness value generated in this manner is therefore the result of a defined measurement procedure, as compared to the more traditional approach where resistance to scratching or cutting has been used to assess the hardness of materials.

The following brief introduction to the mechanical methods employed in the present study explains how the hardness values were obtained. The indenter tip, usually made of a very hard material (e.g. diamond), is pressed to cut into the material surface with a pre-specified applied force (Fig. 1). During the test, the force is loaded and unloaded, and then the entry depth of the tip h is recorded along with the force by a data acquisition system. Based on the recorded data (Fig. 2), the Young's modulus and hardness of the surface are calculated.

Before proceeding any further it is essential to point out that stones are heterogeneous (multi-phase) materials, unlike more homogeneous materials such as steel or aluminium. The influence of microstructures is therefore important to any understanding of material property variability when dealing with natural, unprocessed materials like stone. The property distribution of particles that characterize stone tool raw material must be tested to fully investigate the nature of trace wear development. Tests must therefore be conducted on a micrometer or smaller scale. Nanoindentation testing is one of the modern technologies best suited to investigate these properties.

4. Methods

We used a diamond Berkovich tip indenter (Fig. 3) with a Triboindenter from Hysitron Inc. (Fig. 4), which is specifically designed to conduct nanoindentation tests to obtain the average value and distribution of material properties.

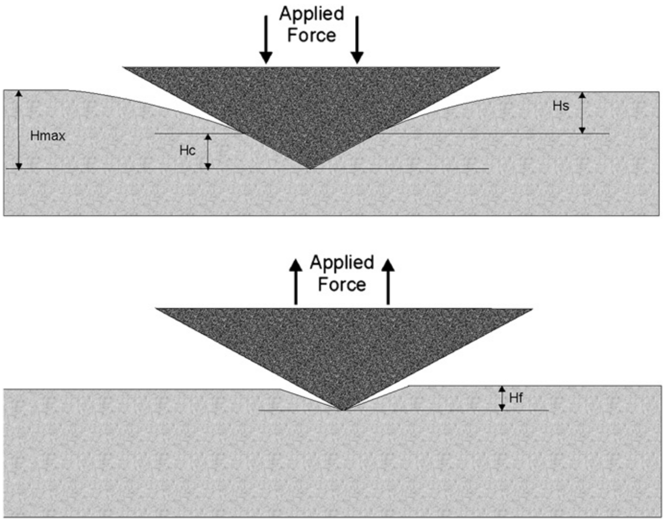


Fig. 1. Schematic of an indentation test: loading followed by unloading.

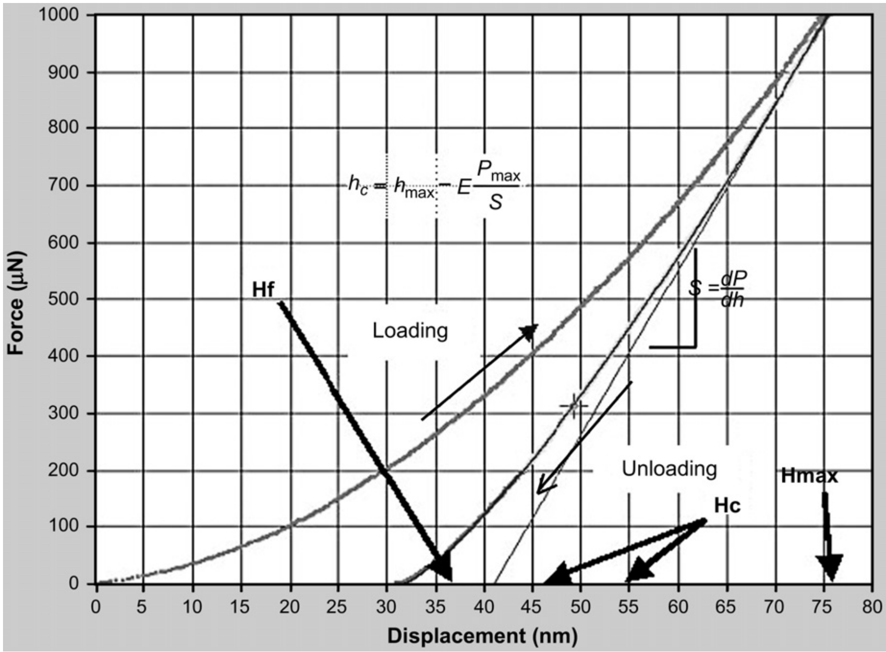


Fig. 2. A typical loading and unloading process during indentation.

The authors studied samples of San Juan Fossiliferous Chert, Brushy Basin Chert, Yellow Silicified Wood, and Morrison Undifferentiated Gray Chert, all materials from the American Southwest, often found on Archaic sites in the Four Corners area (Schutt, 1997a, b). Four samples, one of each material, were cut and polished under identical conditions. Each test sample was tested with six different applied loads ranging from 1000 to 6000 μN and at 10 different locations on its surface. This resulted in a total of 60 indentations per sample and a total of 240 indentations overall. Since these materials are considerably more heterogeneous than those traditionally tested with the Triboindenter it was decided it would be useful to make multiple indentations in order to more fully assess sample variability and more reliably identify significant differences between the samples. The specific locations were selected at random in an effort to collect as representative a set of data as possible.

The four samples were cut from larger nodules using a diamond-tipped saw and were ground down to 11 × 11 × 6 mm in size using diamond-tipped grinding wheels ranging in grit from 80 (which reduces maximum range of surface irregularity to 160 μ) down to 320 (35 μ). These steps were carried out, while maintaining as close to absolute parallelism as possible between the bottom and top surfaces on each sample. Each sample was then lapped, i.e. further ground, using a silicon carbide powder mixed with water that ranged in grit from 800 (10 μ) to 1000 (5 μ). This was done to ensure removal of all grind marks and unevenness on the samples.

For the final round of polishing each sample was mounted onto a glass slide using a cyanoacrylate-mounting medium to

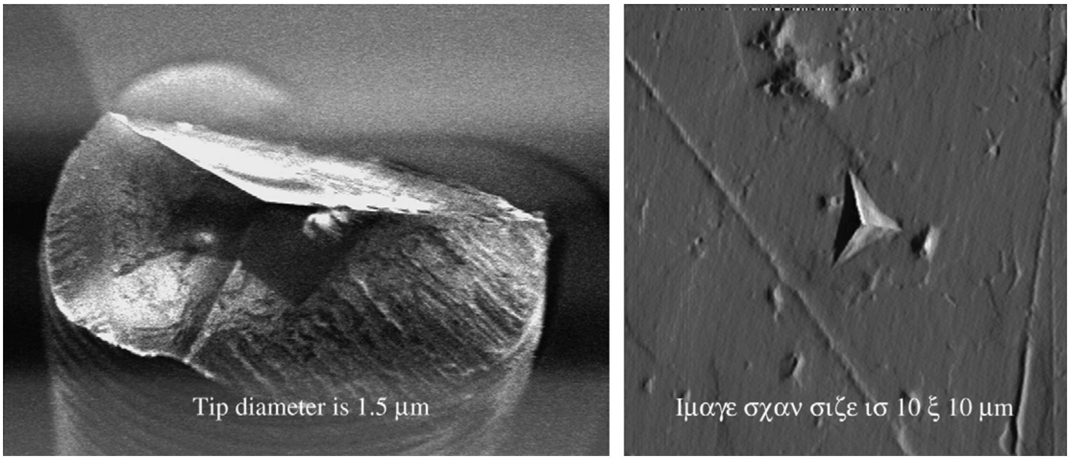


Fig. 3. The Berkovich tip and resulting indentation.

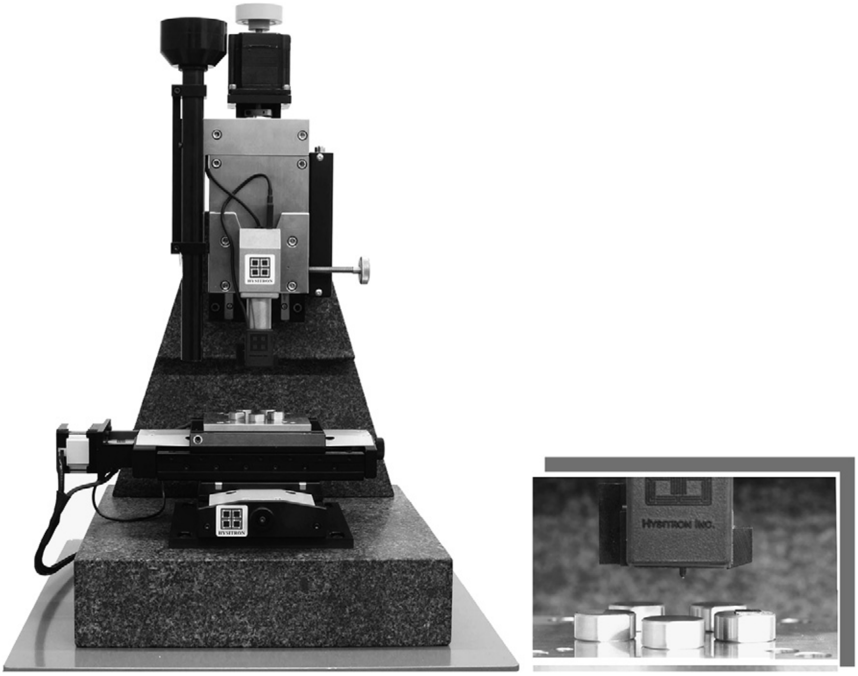


Fig. 4. The Triboindenter (Hysitron Inc.).

secure the sample in place. The slides were then placed in Logitech PM2A polishing machines that use alumina oxide combined with water and ethanol glycol as a polishing agent. The polishing was done in a series of stages that include agent grits of 5 μ for 15 min, 3 μ for 30 min, 1 μ for 60 min and 0.5 μ for 20 min. The last stage was followed by one additional round of polishing with 0.3-μ grit diamond paste that lasted for 20 min. All polishing was done on a substrate of pellon (a polishing cloth material) and resulted in mirror finish on the top surface of each sample. The samples were then soaked in acetone for 24 h to dissolve the cyanoacrylate holding them to the glass slides. This was followed by a 20-min ultrasound bath in distilled water to remove any remaining residues (George Panagiotidis, 2005 pers. comm.). Once sample preparation was complete they were affixed to the Triboindenter stage for indentation (Fig. 5).

Along with the hardness data, the Triboindenter also provided topographical scans and information regarding the level of high surface smoothness on the prepared samples. This provides a preliminary assessment of microtopographic variability.

A three-stage experimental program was carried out using the four raw materials tested with the indenter. The experiments analysed here involved the scraping of dry ungulate hide. While performing these activities all experimental flakes were used in the same manner. This included maintaining a constant implement to working surface angle of 45° as well as performing all activities on the same contact materials throughout all stages of the experimental program. This was done to maximize consistency of wear production to facilitate comparisons between raw material types. The first stage of the experiment involved 10 min of continuous use, the second an additional 20 min resulting in a total of 30 min of use, and the third a further 30 min resulting in a final total of 60 min of use per activity per flake.

The experiments were carried out with the assistance of five undergraduate students. While due consideration was given to the idea of having the same person conduct the same experiments throughout the entire program, it was decided to let each person perform a range of tasks in order to more accurately replicate the sort of variability inherent in the archaeological record. All tools were hand-held, therefore unhafted, since the short-term production and use of their archaeological counterparts would have made hafting an inefficient and impractical enterprise. Hides were extended over sheets of plywood and nailed down to restrict their movement during use. They were then placed on the laboratory floor where scraping was carried out.

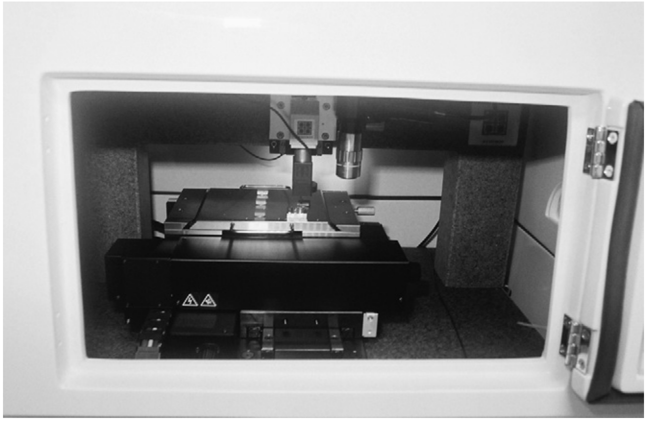
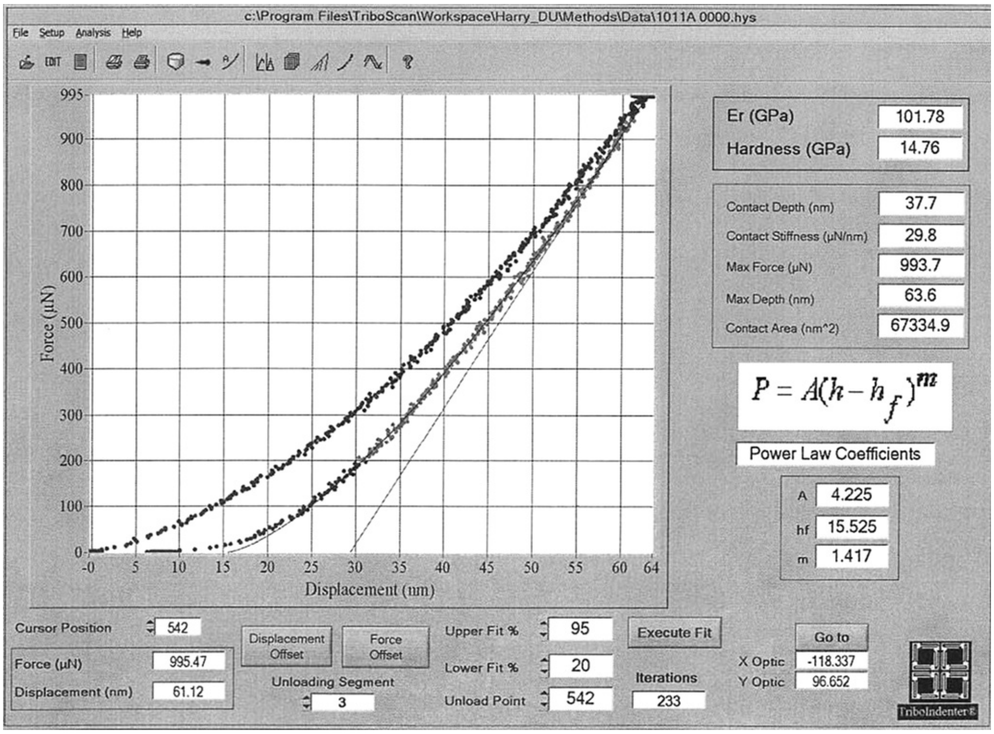


Fig. 5. The four $1 \times 1 \times 0.5$ cm samples on the Triboindenter stage, one directly under the indenter tip and the other three lined up on the left.



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Er (GPa): 101.78
Hardness (GPa): 14.76

Contact Depth (nm): 37.7
Contact Stiffness (μN/nm): 29.8
Max Force (μN): 993.7
Max Depth (nm): 63.6
Contact Area (nm^2): 67334.9

$$P = A(h - h_f)^m$$

Power Law Coefficients
A: 4.225
hf: 15.525
m: 1.417

Cursor Position: 542
Force (μN): 995.47
Displacement (nm): 61.12
Unloading Segment: 3
Upper Fit %: 95
Lower Fit %: 20
Unload Point: 542
Iterations: 233
X Optic: -118.337
Y Optic: 96.652

Fig. 6. Output from Hysitron software for a single indentation on SJF (1011).

Following each stage, i.e. after 10, 30 and 60 min of use, each experimental flake was subjected to a multi-step cleansing regimen to maximize trace wear visibility to allow for accurate and reliable analyses of wear accrual patterns. This consisted of initially washing each tool in warm water with mild detergent to remove all visible residues and debris. This was followed by soaking each flake for 30 min in a sonic bath of 30% NaOH solution using a Brandon 1510 Sonic Cleaner. Lastly, each tool was rinsed with distilled water to remove any contamination associated with previous handling of the implements. Initially thought was given to including a wash with 10% HCl solution, but since in the prehistoric past adhering residues would have acted as a tertiary abrasive agent the decision was made to omit this step to keep in line with the naturalistic mandate of this study.

Following cleaning the tools were placed in a Hummer VI Sputtering System sputter coater so that they could be coated with a gold–palladium alloy to maximize the conductivity of the sample. Increased conductivity essentially eliminates charging of surface electrons, resulting in much clearer image generation. Each tool was kept in the sputter coater for 5 min per side to ensure it was completely coated with the alloy to a thickness of three nanometers. Each flake was then examined at 100× magnification using JEOL JSM-840A and Hitachi 4200 Variable Pressure scanning electron microscopes. The resulting images were imported into the ClemexVision Professional Edition (Version 3.5) digital image analysis software package for quantitative analysis of edge modification and overall wear invasiveness (Lerner, n.d.).

5. Results

Fig. 6 is an example of a typical output screen generated by the Hysitron software. Notable differences were observed in the relative hardness of each raw material. From hardest to

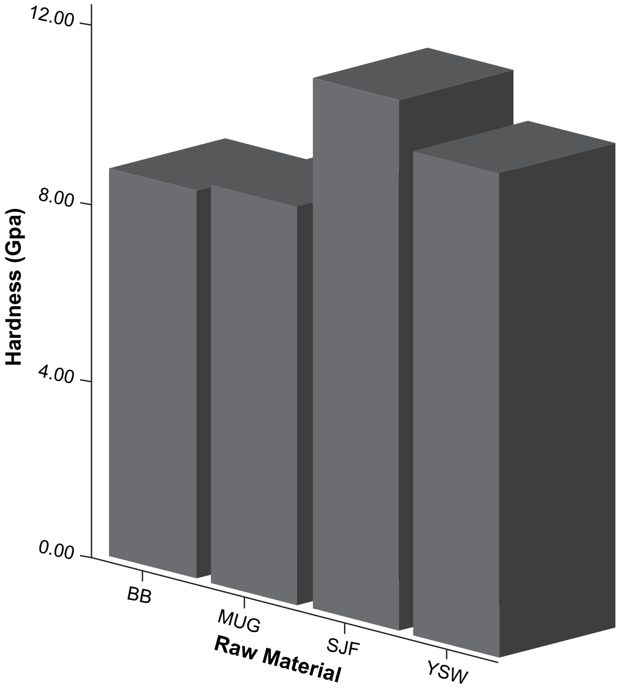

Fig. 7. Hardness by raw material.

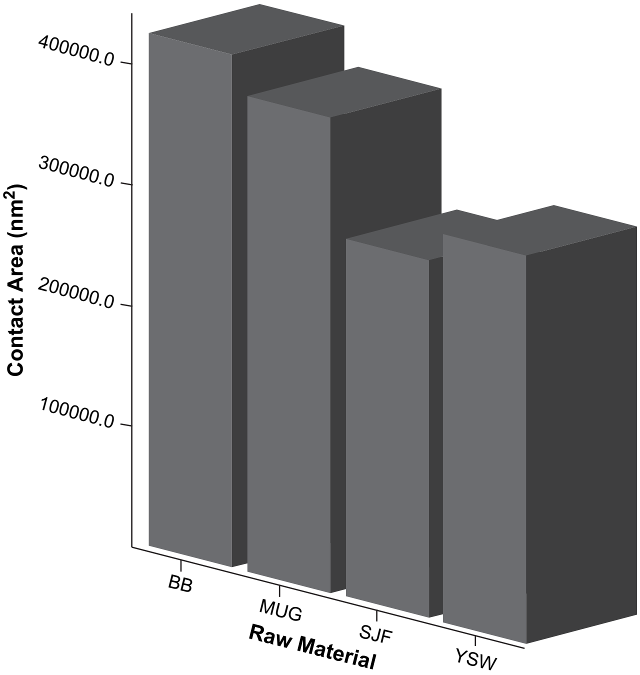

Fig. 8. Contact area by raw material.

softest the four raw materials ranked as follows: San Juan Fossiliferous chert (SJF) with a mean hardness of 12.08 GigaPascals (GPa), Yellow Silicified Wood (YSW) with 11.02 GPa, Morrison Undifferentiated Gray chert (MUG) with 9.08 GPa, and Brushy Basin chert (BB) with 8.83 Gpa (Fig. 7).

A lower GPa value indicates a relatively soft material and a higher value a relatively hard material. These results were corroborated by data generated concerning indentation or contact area and maximum depth of penetration. Both of these measures reflect the same hardness ranking described above. Harder materials, SJF and YSW, are characterized by indentations with smaller contact areas and shallower depths, whereas softer materials exhibit indentations with larger contact areas and greater depths (see Figs. 8 and 9, respectively). Fig. 10 shows this relationship via images of single indentations on each raw material captured with the integrated atomic force microscope on the Hysitron Triboindenter. These indentations were all made with the same applied load of 6000 μN, thus effectively conveying the influence of material hardness on the extent of surface deformation under equivalent conditions.

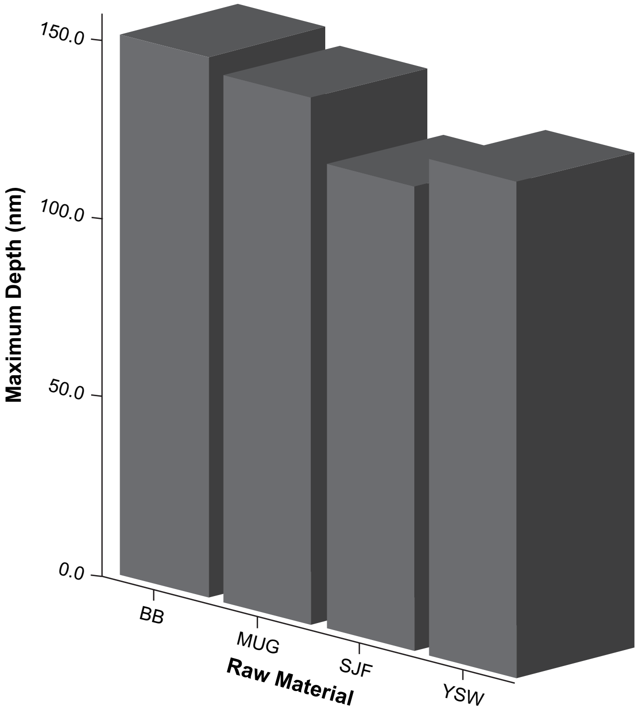

Fig. 9. Maximum depth by raw material.

Such differences in material hardness, not surprisingly, have significant implications for interpreting archaeological wear traces. Most importantly the relative hardness of a given material will contribute to determining the rate at which wear will accrue during the course of tool use. How this influence is exerted is complex and is beyond the scope of this paper. However, data regarding the mean variance of hardness values for each raw material have been generated and compared as a preliminary evaluation of this complexity. The variance data was viewed as a way to gauge both material heterogeneity and the complexity of the role of hardness in use-related wear accrual.

To be as thorough as possible, variance was assessed first in terms of applied load and then according to indent location, and as Figs. 11–14 illustrate the results in each instance were quite similar. In terms of absolute hardness values, as measured in GPa, SJF exhibited the most variance in these values, followed by BB, MUG and YSW, which showed the least (Figs. 11 and 12). When looking at mean variance as a percentage of the overall range of hardness values, BB actually has a higher variance than SJF, with MUG and YSW rounding things out in decreasing order (Figs. 13 and 14). The mean variance in hardness values for these four raw materials shows clear differences, strongly suggesting that increasing variance is clearly indicative of increasing material heterogeneity.

The scraping of a dry ungulate hide serves as a brief example of how material properties affect patterns of use-related wear accrual. The degree of edge rounding resulting from this activity tended to be most prominent on MUG, the second softest of the four materials, and tended to be least pronounced on YSW, the second hardest material (Fig. 15). SJF and BB exhibited intermediate degrees of edge rounding, even though the SJF scraper experienced edge collapse during the second stage of the experiment. In terms of overall wear invasiveness YSW consistently developed wear more uniformly than any of the others, that is wear covered a greater percentage of the overall surface microtopography. MUG tended to develop the most invasive wear except after the last stage of the experiment, which resulted in YSW having the most invasive wear. BB, the softest of the four materials, ultimately accrued the least amount of invasive wear while SJF accrued the least amount of wear through the first and second stages of the experiment (Fig. 16).

It is clear that patterns of wear accrual are determined by a combination of material surface hardness and surface

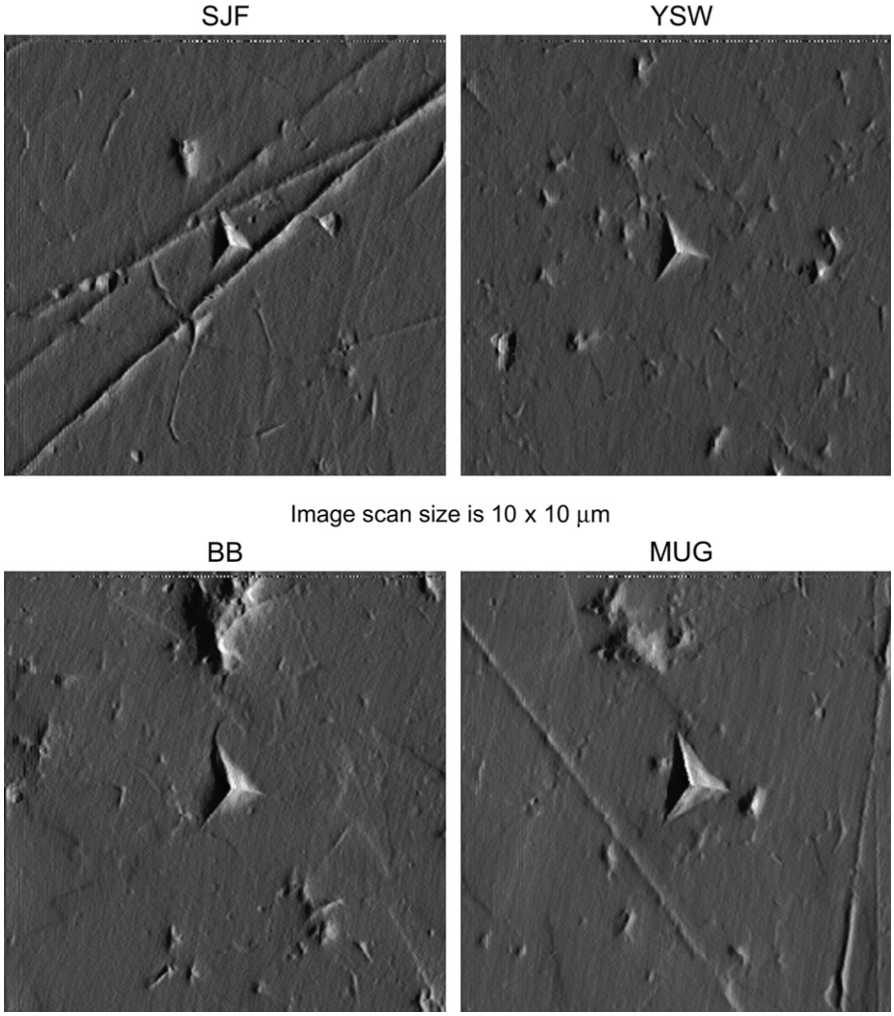

Fig. 10. Indentations on each raw material under maximum load.

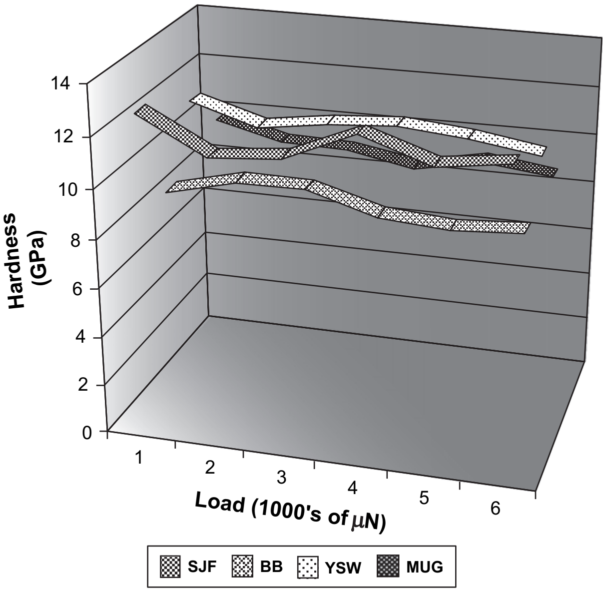

Fig. 11. Variance in hardness by load.

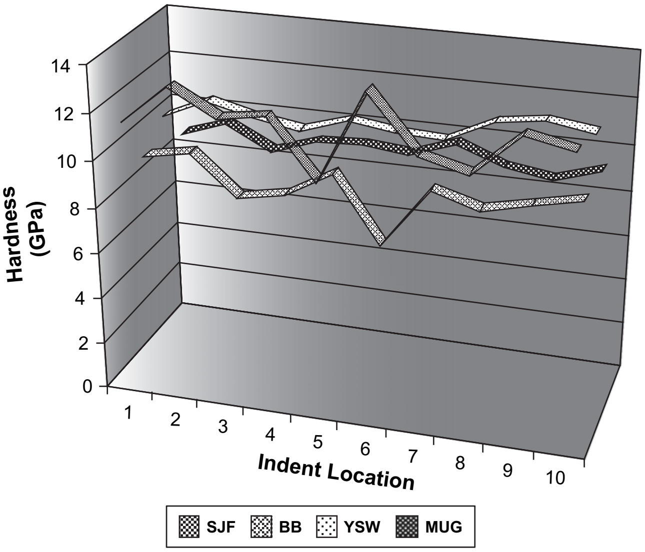

Fig. 12. Variance in hardness by indent location.

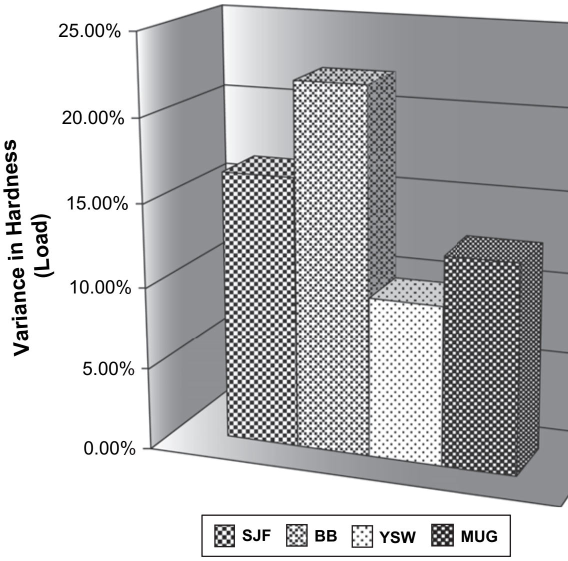

Fig. 13. Mean variance in hardness by load.

microtopography. In the case of the relatively hard YSW, it's greater surface regularity promoted the development of more homogeneous and ultimately more invasive wear, as opposed to BB with its relative softness being tempered by its more irregular surface characteristics.

6. Discussion

The relationship between hardness, surface roughness, and wear accrual is highly dynamic and complex. The results presented here highlight the inherent complexity of the role of raw material physical properties during use and emphasize the need to avoid broad generalizations regarding how certain wear attributes are diagnostic of certain activities. They also suggest that in addition to differences between material types, we need to consider the possible effects of inherent variability within a single material type. The indentation of each material sample under different loads and at 10 different surface locations provided data representative of both the samples as wholes and of their internal variability. While quantifiable differences between the four stones were found, the results also highlighted the fact that none of these materials is perfectly homogeneous. This demonstrates that a given tool will not accrue wear evenly across its entire surface, and that this must also be considered when interpreting wear traces in behavioural, and ultimately cultural, terms.

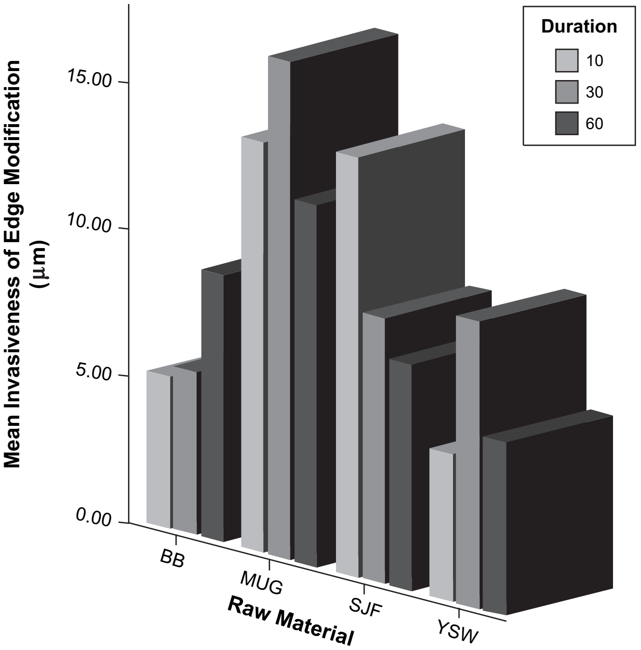

Fig. 15. Edge modification due to scraping dry hide by raw material.

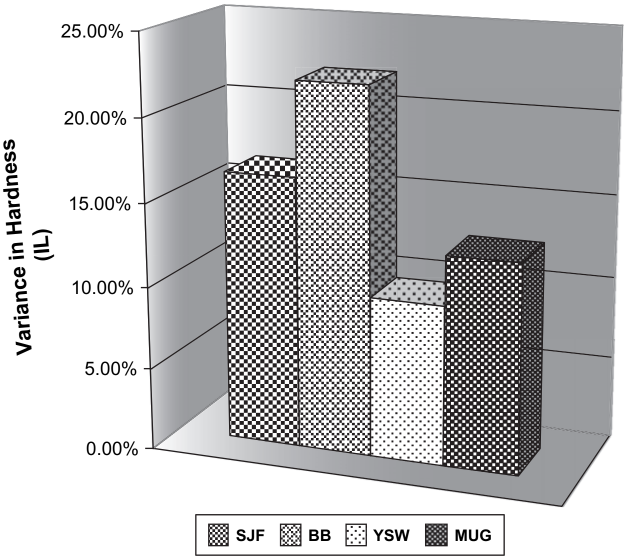

Fig. 14. Mean variance in hardness by indent location.

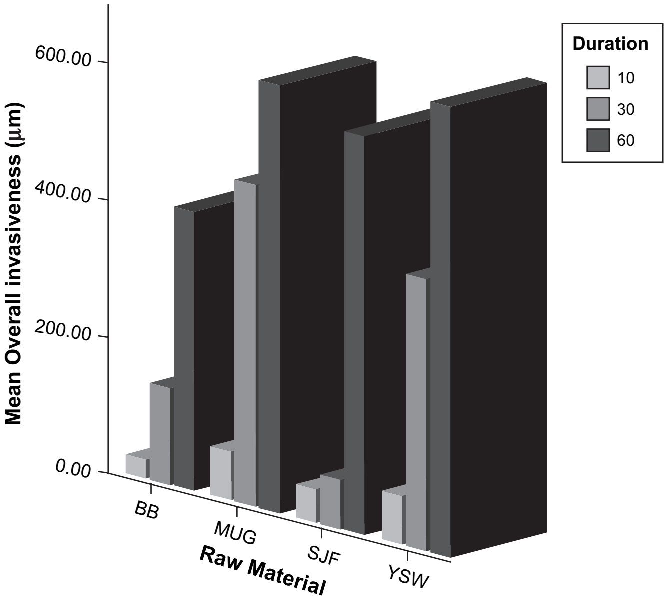

Fig. 16. Overall wear invasiveness due to scraping dry hide by raw material.

Although most crystalline silica is characterized by a hardness value of seven on the qualitative Moh's scale, the results described above demonstrate that from one variety to another significant differences do exist and can be detected. This has important implications for lithic use-wear analysis and demands careful scrutiny when it comes to making inferences on the basis of wear form and extent. A more thorough understanding of differences in material hardness and how they affect wear development is essential to forming more reliable interpretations of function. This is particularly critical when dealing with a lithic archaeological record comprised of several different raw materials. The differences between the four materials under consideration influenced the development of prehistoric wear traces to the degree that extent of wear alone cannot be used to determine tool function, much less use intensity.

But hardness is only part of the equation for the relationship between material properties and wear accrual rates. Microtopographic variability or surface heterogeneity also plays an important part in how wear develops. The Triboindenter tests yielded some data regarding sample surface roughness that offer some preliminary insight into how respective surface characteristics of each raw material may influence wear accrual over time. It is important to note that despite the extensive polishing of the samples, differences in surface microtopography were still detectable. Since all samples were prepared according to exactly the same set of procedures (described above) these differences can only be attributed to inherent differences in these materials. Thus, it is absolutely essential that we include this variable in our consideration of how the physical properties of raw materials may influence use-related wear accrual.

The sample surface data generated in this study indicate that YSW exhibits the least amount of microtopographic variability. At the other end of the spectrum BB is the most heterogeneous or variable, and SJF and MUG are intermediate in order of decreasing sample surface variability. Figs. 17 and 18 are bar graphs of measurements of average surface roughness and peak-to-valley distances, respectively. The data presented in Fig. 17 are mean values for randomly selected areas on the sample surfaces, while Fig. 18 illustrates the maximum difference in microtopographic height between the lowest and highest points across the same areas depicted by Fig. 18.

Fig. 19 further illustrates these differences by comparing BB and YSW, the two extremes, in terms of three dimensional plots of sample surface roughness. A forthcoming paper deals with the application of GIS analysis to evaluating use-related changes in tool surface microtopography over time as a function of raw material properties.

7. Conclusion

The last 40 years have seen considerable progress in developing our understanding of use-wear evidence in both archaeological and materials science terms. This progress is in part due to increasing methodological overlap between these two fields of research, but as the present study can attest, a great deal more work still awaits if either discipline is to realize the full potential of this relationship. Materials science can benefit from greater insight into the complex dynamics of wear generation on heterogeneous materials, and archaeology can continue to develop more reliable methods of assessing wear traces once the influence of raw material properties is more fully understood.

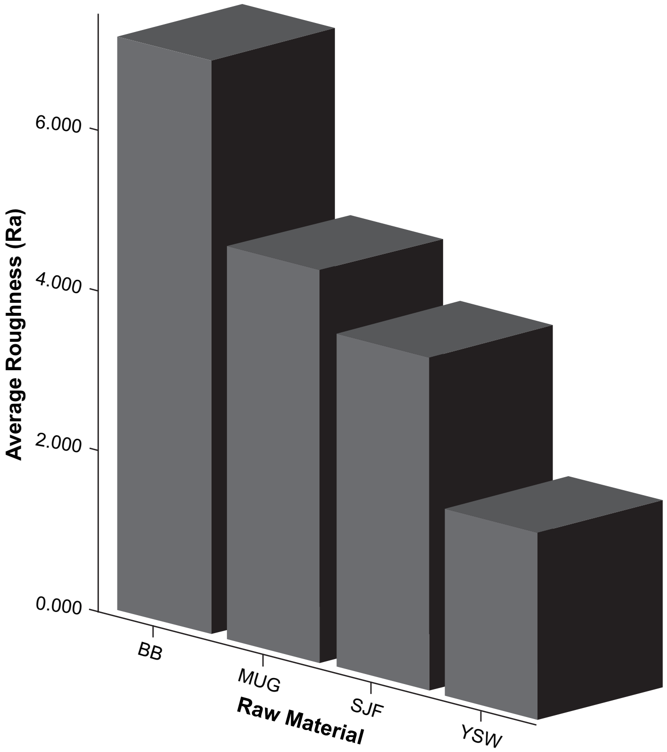

Fig. 17. Average roughness by raw material.

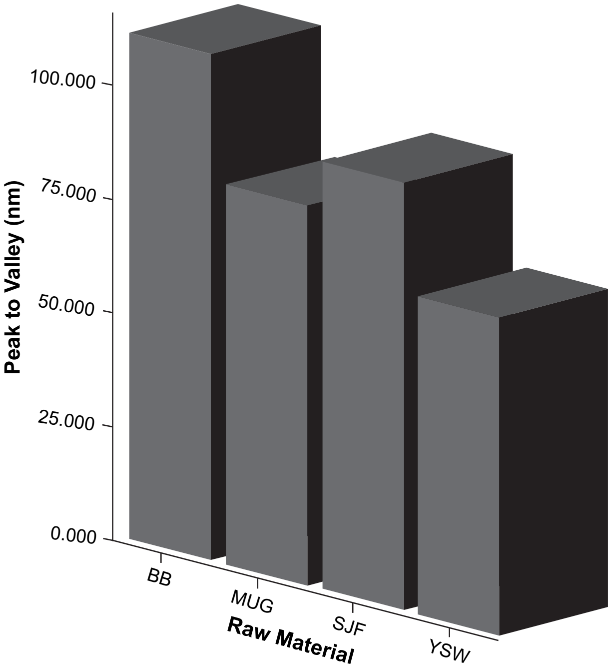

Fig. 18. Peak to valley by raw material.

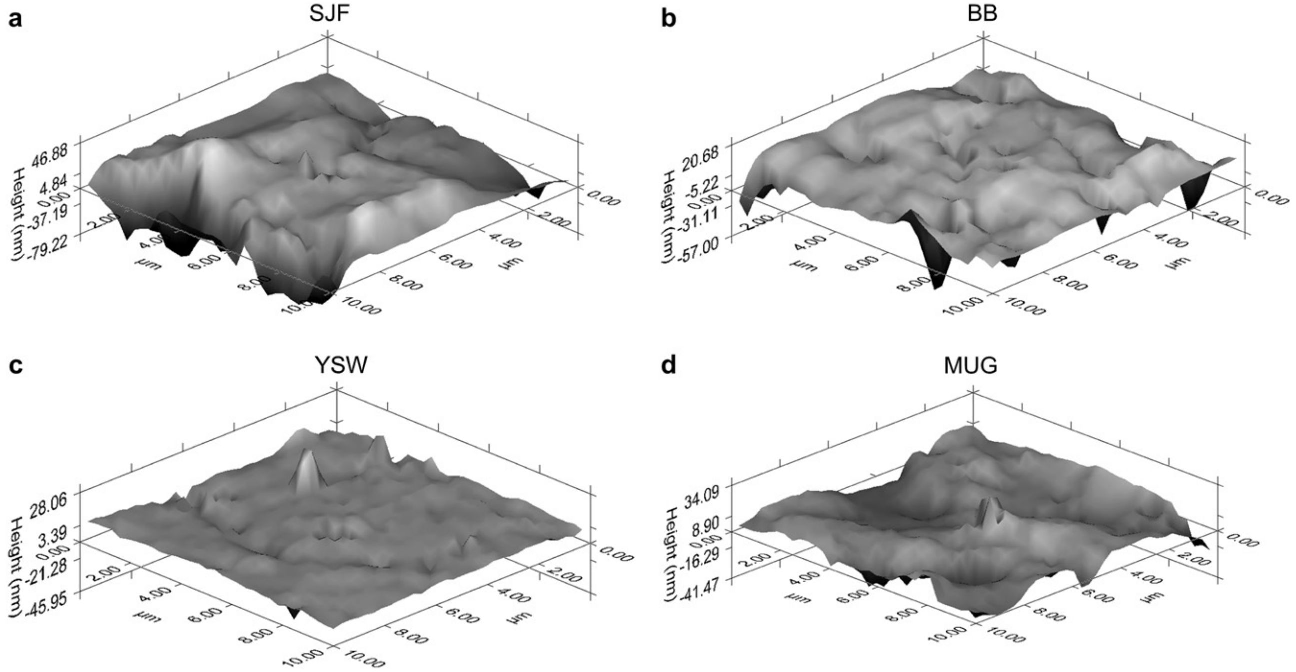

Fig. 19. 3-D plots of sample surface roughness for SJF (a), BB (b), YSW (c) and MUG (d).

Acknowledgements

The authors would like to thank George Panagiotidis for his exceptional work in preparing the hardness testing samples. Sample preparation was made possible through an internal SSHRC grant provided by McGill University. We would like to acknowledge that the work of Xiangdong Du and Dr. Martin Ostoja-Starzewski has been made possible through NSERC and the Canadian Research Chairs Program. We also thank Drs. Peter R. Norton and David J. Munoz-Paniagua of the Department of Chemistry at the University of Western Ontario for generously allowing us access to their Triboindenter lab, and their invaluable time and advice. A debt of gratitude also goes to Dr. Michael Bisson of the Department of Anthropology at McGill University for providing us with the experimental tools we used in this study, and to Roger Moore of the Chaco Culture National Historical Park for generously supplying the lithic raw materials necessary for carrying the experiments. We would also like to acknowledge the contributions made by Jennifer Bracewell, Jennifer Dickson, Deanna Dytchkowskyj, Colin Nielsen and Joanna Rowell through their invaluable participation in the experimental program.

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