Use-wear analysis of non-flint stone tools using DIC microscopy and resin casts: a simple and effective technique

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Abstract: By means of example taken from experiments and archaeological study of stone tools from Upper Palaeolithic sites from Portugal (Estremadura and Côa Valley), the goal of this paper is to highlight the accuracy of the Differential Interference Contrast microscopy associated with epoxy casts, in use-wear analysis of highly reflective surfaces of non flint stone tools, such as quartz, quartzite and rock crystal.

Résumé: A travers des exemples de supports lithiques expérimentaux et du paléolithique supérieur du Portugal (Estremadura et Vallée du Côa), cet article a pour objectif démontrer l’efficacité de la microscopie à contraste interférentiel associée à des répliques en résine époxy, dans la l’analyse tracéologique de surfaces réfléchissantes de matières premières lithiques alternatives au silex, telles que le quartz, le quartzite et le cristal de roche.

Resumo: Através de exemplos de suportes líticos experimentais e do Paleolítico Superior de Portugal (Estremadura e Vale do Côa), este artigo tem como objectivo sublinhar a eficácia da microscopia com contraste interferencial associada a réplicas em resina no estudo traceológico de matérias-primas líticas alternativas ao sílex, tais como o quartzo, o quartzito e o cristal de rocha.

Introduction

The last two decades have seen a shift in use-wear research, greatly due to the progress of modern technology and the development of sophisticated techniques. Since the previous work focused upon identifying the mechanisms of polish formation, many studies have examined new techniques to derive tool use-wear and contributed to the systematisation of the use-wear analysis. The majority of these studies have centred upon flint and the method is well established. However the situation is quite different concerning stone tools made on non flint raw materials. Tools made on quartz, quartzite, rock crystal, obsidian for example, have been neglected, often excluded from use-wear studies due to the physical properties of these raw materials, rendering the observation under microscope highly difficult, and leading to a lack of knowledge of these kinds of rocks.

In the past few years there has been considerable innovation in use-wear methods for non flint raw materials along with the development of several original analytical techniques to assess between different wear patterns. Recent use-wear studies of non flint raw materials focused in developing alternative methods in order to overcome the analytical constraints imposed by the physical properties of these raw materials. Unfortunately, in spite of the use of highly sophisticated equipment these techniques are still not entirely satisfactory. Indeed,
the implications of the results, mainly limited to experimental data, are presently unclear due to the restricted samples used and the lack of archaeological application. Further to these problems, the sophisticated equipment required by these techniques is not always easily available.

By means of example taken from experimental and archaeological study of stone tools from Upper Palaeolithic sites from Portugal, the goal of this paper is to highlight the accuracy of the Differential Interference Contrast Microscopy, also known as Nomarski contrast, in the study of highly reflective raw materials such as quartz, quartzite and rock crystal. Moreover, it is shown how the high-resolution epoxy casts of the edges also increases the visibility of use-wear on these kinds of raw materials.

Background

Since the previous work centred upon the nature of use-wear formation, many studies, designed to search for analytical procedures in order to establish use-wear patterns, have contributed to the systematisation of the use-wear approach on lithic materials (Kamminga, 1979; Levi-Sala, Tringham et al., 1974; Odell, 1979; Keeley, 1980; Plisson, 1985; Anderson-Gerfaud 1981, Mansur-Franchome, 1986). The majority of these studies were centred upon flint. This is related with the fact that non flint raw materials are particularly difficult to study under microscope. Their physical properties, such as a grosser granular and highly reflective surface, enable use-wear analysis with bright field microscopy, commonly used to flint.

Along with the progress of modern technology, a number of studies have been carried out, centred on the development of advanced analytical methods in order to determine important factors that are of influence to stone tool use-wear on non flint raw materials. In the past few years specific analytical techniques were developed, some using highly sophisticated equipment like the SEMi (Broadbent, Knutsson, 1975; Knutsson, 1986, 1988a, 1988b, 1989; Sussman, 1988; Beyries, 1982 ; Plisson, 1985, 1986 ; Hurcombe, 1992; Philibert, 1993; Younping, knutsson, 1995; Pignat, Plisson, 2000 ; Byrne, Ollé, Vergés, 2006; Clemente, 1997), which uses electrons to create an image with increased depth of field in contrast to the reflected light used in the metallurgical microscope. More recently, the CLSMi (Derndarsky e Ocklind, 2001) dealing with the analysis of the subsurface damage on quartz tools by dying them with fluorescent colour and scanning them, generating stacks of optical sections taken at successive focal planes that produce a three dimensional view of the artefact. Other examples of innovative methods are the nanoidentation technique which consists on measuring average value and distribution of material properties such as surface hardness and roughness with a diamond Berkovich tip indenter (Lerner et al., 2007) and the XRD (X-ray diffraction) of raw material composition and microstructure (Yonekura, K.et al., 2008). These studies, often limited to experimental programs or with little archaeological application, are still not entirely satisfactory. Further to this, some of the analytical equipment used is not easily available.
The Differential Interferential Contrast microscopy

The best results to examine the surfaces of non flint implements were obtained using the differential interferential contrast (DIC). This method, developed by G. Nomarsky, which resolution is similar to the SEM, but performed in a simpler manner, was first outlined by K. Knutsson (1988b) and also used by H. Plisson (Pignat et Plisson, 2000).

Indeed, bright field microscopy proved inadequate to composition and structure of non flint stone tools, because it cannot sense low-amplitude defects of the polished surfaces. The nature of the various crystal phases and textures within polycrystalline materials significantly affects their mechanical and electrical properties. Structural defects such as micro-defects, stacking faults, dislocations, twins, small-angle and large-angle boundaries, affect the mechanical, electrical and optical properties of single crystal and polycrystalline materials.

Slopes, valleys, and other discontinuities on the surface of the specimen create optical path differences, which are transformed by reflected light DIC microscopy into amplitude or intensity variations that reveal a topographical profile. Unlike the situation with transmitted light and semi-transparent phase specimens, the image created in reflected light DIC can often be interpreted as a true three-dimensional representation of the surface geometry, provided a clear distinction can be realized between raised and lowered regions in the specimen.

When compared to the typical configuration employed in transmitted light microscopy, the critical instrument parameters for reflected (or episcopic) light differential interference contrast (DIC) are much simpler, primarily because only a single birefringent Nomarski or Wollaston prism is required, and the objective serves as both the condenser and image-forming optical system. Because of the dual role played by the microscope objective, a Nomarski prism interference pattern projected into the objective rear focal plane is simultaneously positioned at the focal plane of the condenser illuminating lens system.

A schematic cutaway diagram of the key optical train components in a reflected light differential interference contrast microscope is presented in Figure 1. Illumination generated by the light source passes through the aperture and field diaphragms in a vertical (episcopic) illuminator before encountering a linear polarizer positioned with the transmission axis oriented East-West with respect to the microscope frame. Linearly polarized light exiting the polarizer is reflected from the surface of a half-mirror placed at a 45-degree angle to the incident beam. The deflected light waves, which are now traveling along the microscope optical axis, enter a Nomarski prism housed above the objective in the microscope nosepiece where they are separated into polarized orthogonal components and sheared according to the geometry of the birefringent prism.

Acting in the capacity of a high numerical aperture, perfectly aligned, and optically corrected illumination condenser, the microscope objective focuses sheared orthogonal wavefronts produced by the Nomarski prism on the surface of an opaque specimen.
Reflected wavefronts, which experience varying optical path differences as a function of specimen surface topography, are gathered by the objective and focused on the interference plane of the Nomarski prism where they are recombined to eliminate shear. After exiting the Nomarski prism, the wavefronts pass through the half-mirror on a straight trajectory, and then encounter the analyzer (a second polarizer) positioned with the transmission axis oriented in a North-South direction. Components of the orthogonal wavefronts that are parallel to the analyzer transmission vector are able to pass through in a common azimuth, and subsequently undergo interference in the plane of the eyepiece fixed diaphragm to generate amplitude fluctuations and form the DIC image. Formation of the final image in differential interference contrast microscopy is the result of interference between two distinct wavefronts that reach the image plane slightly out of phase with each other, and is not a simple algebraic summation of intensities reflected toward the image plane, as is the case with other imaging modes. This technique is ideal for use-wear studies on highly reflective materials because it affords a high resolution, contrast and three-dimensional view of microtopography superior to conventional binocular microscopes commonly used to examine use-wear traces (fig.2).
Moreover it can also be applied to flint implements, since it improves greatly the quality of digital microphotographs. Given its effectiveness it can appears surprising why so much effort has been done in the development of sophisticated and complex analytical techniques that are at the end often less satisfactory.

All the edges and surfaces of experimental and archaeological stone tools were analysed under a reflected light microscope (Olympus BH, 100x e 200x) with DIC and photomicrographs were taken using a digital camera Nikon 4500 at 50, 100 and 200x magnification before and after the experiments.

**Edges high-resolution epoxy casts procedure**

Firstly used by archaeozoologists to study surface bones modification (Selvaggio, 1994) this method can easily replicates and document microscopic use-wear features on lithic surfaces. Its accuracy on lithics was recently described by W. Banks and M. Kay (2003), but only from the point of view of its application on chemical weathered flint surfaces. They used it on a restricted sample (two artefacts) of patinated Middle Palaeolithic stone tools from the site Solutré Village, advocating that it allows to determine use-wear analysis of weathered surfaces. Beside the fact that was a restricted sample and that the capacity of epoxy cast in overcoming post-depositional surface modifications can be discussed, their work was centred on flint.

Before casting, artefact surfaces are cleaned of any adhering sediment and oils with liquid detergent and alcohol. The mould of the edge is made with polyvinylsiloxane gel manufactured by Coltene-Whaledent president light body. This product is currently used by dentists. It comes in paired tubes. The gel reproduces features visible up to 10,000X magnification and maintains its integrity. The artefacts surface and adjacent edges are covered by this gel, producing a mould. The gel should harden for three or 4 minutes after which time the artefact can be removed. To make the positive cast a mixture of epoxy, epoxy hardener is slowly poured into the mould that was previously placed in air hardening modelling paste. The mixture takes few hours to get hardened, and can easily be removed from the mould (fig.3). Finally, the resin can be analysed directly under microscope.
A dark pigment can be stir into the epoxy hardener mixture until it becomes opaque, in order to get resembled unweathered flint artefacts, affording detailed photomicrographs. High-resolution epoxy casts of the edges of non-flint stone tools improves greatly the visibility of use-wear on highly reflective tool surfaces and also the quality of digital microphotographs (Fig.4).

Non flint stone tools use-wear: experimental results

The method described was developed in the scope of a research project centred on the restitution of human economical and social strategies during Upper Palaeolithic in Portugal through stone tools use-wear analysis.

Taking into account that Portuguese Upper Palaeolithic lithic industries are highly composed of quartz, quartzite and rock crystal, and since these kinds of raw materials do not develop use-wear in the same manner as flint, experimental work was specifically undertaken in order to determine use-wear features on non flint stone tools that will be compared with archaeological record.

Experiments described in this paper are the first from a more extensive experimental program, designed to test a
variety of rocks, in particularly quartzite. A variety of implements (blades, bladelets and flakes), were replicated on quartzite, quartz and rock crystal (n=33), all of them hand yielded, except in the case of rock crystal bladelets which were used side hafted in a handle of wood and in spear shafts.

Thirty three stone tools were knapped (fig.5):
- Flakes unretouched on quartz (n=5) and on quartzite (n=5) ;
- Endscrapers made on quartz (n=2) and on quartzite (n=4)
- Seventeen bladelets on rock crystal, further inserted in two pieces of wood (7 bladelets side hafted in a thick handle and 10 in a thinner one).

Tools were used retouched and unretouched to perform different activities in order to recognise use-wear features on these rocks which were compared to archaeological record. Projectile, butchery and hide processing experiments were executed in an extensive manner, including all technical tasks comprised in the operative chains of these activities. Bone, fish and wood were tested more punctually and into specific working motions (cut, scrape).

Each tool, experimental and archaeological, was used for a single activity in order to better isolate and distinguish use-wear obtained. After use, tools were cleaned with mild detergent and rinsing with alcohol. Then, they were examined under a reflected light microscope standard with differential interference contrast (DIC) following the standard procedures used in use-wear analysis. Tools surfaces and edges were photographed before and after experiments (fig.6).
In experiments dealing with processing activities, all tools were used for more than half an hour, with the exception of the bladelets side hafted in a wood handle, which were used much longer.

**Projectile points**

Taken into consideration that backed tools, made on rock crystal and quartz, were found in the studied Upper Palaeolithic contexts, and that these kind of tools are thought to be elements of throwing weapons, experimental spears, with hafted backed points, were produced and hurled by hand against a roe deer. Seventeen bladelets were hafted in three spear shafts of wood of one meter long, fixed with a mixture of resin, bee hoax and ochre, and feathered. Each spear was shot repeatedly until the point or the shaft was damaged (fig.7).

In spite of the shots were executed immediately after the roe deer being killed in order to avoid the natural *rigor mortis* of the carcass, it should be underlined the difficulties for the spear points in getting inside the carcass. This is probably related with the eight of the shaft which was too light and with the projectile delivery mode as well. The use of a spearthrower usually facilitates hitting the target. Only one spear point has penetrated the animal’s hide but hasn’t touched the bone. In this case, the point fractured and remained inside the hide.
**Resulted use-wear**

There were visible fractures that were analysed according to the methodology proposed by Fisher *et al.* (1984), based on morphology of the initiations and terminations. However, tools don’t show any typical projectile fractures like those usually considered diagnostic for projectile function like bending fractures and spin offs. Instead, bladelets show snap fractures which could also be induced by natural phenomena (fig.8).

![Figure 8 – Experimental projectile bladelets with snap fractures.](image)

**Roe deer carcass processing**

The roe deer carcass processing tasks – dismemberment, filleting and tendon removal - were performed using two unretouched flakes (on quartzite and on quartz) and a composite tool made of wood, with side hafted bladelets made on rock crystal (fig.9). All tools were used to cut during more than thirty minutes and remained highly effective. As already outlined by other authors, quartz is especially accurate to butchering, as the cutting capacity of the edge remains the same in spite of time using. The side hafted rock crystal bladelets were particularly effective and much longer, no matter the hardness of the contact materials (meat, hide and tendons).
Figure 9 – Experimental butchering of a roe deer: a) quartzite knapping; b) roe deer carcass; c) dismembering with a flake made on quartz; d) with rock crystal bladelets of a composite tool (Photos J.P.Ruas).

Resulted use-wear

Tools used to dismember and to remove tendons are the most affected by edge scarring, located on both sides of the edge.

Like expected, microscopic use-wear (polishes) related with the contact of bone and meat only develop on quartz and quartzite. On rock crystal implements only striae and scars are visible. For quartzite and quartz, use-wear related with cutting meat is barely recognizable, as it is also noticed by other analysts concerning this kind of activity (Plisson, 1985; Clemente, 1997; Gibaja, 2005). In a global manner, use-wear is not very developed due to the occasional contact of the tool’s edge with the worked material, which is usual in this kind of activity (fig.10).
Figure 10 – Stone edges and surfaces before and after carcass processing (200x): a) polish on quartzite produced by cutting hide; b) polish on quartz after cutting meat; c) polish on quartzite produced by cutting meat; d) striations on rock crystal after the carcass dismemberment (Photos J.P.Ruas, micrographs M.de Araujo Igreja).
Hide processing

This experiment consists in the hide processing of a roe deer. The technique used followed the same documented through ethnographic studies among actual and sub-actual populations such as the work of B. Robbe (1975) on the Esquimos from Greenland where several tasks are described (fig.11).

First, the hide was laced on the ground with vegetal rope on a wood frame, in which the lacing holes were made by using a quartzite borer; then we have proceed to the hide fleshing by eliminating adhered residues with unretouched flakes and endscrapers made on quartzite and quartz. The hide was soaked with abrasive substances, a mixture of ochre and ashes in order to stretch and staining the hide and scraped continuously with endscrapers made on quartz and quartzite. The fur was easily removed by hand. The hide was scraped again with the compound. Finally, the hide was removed from the frame by cutting it with unretouched quartzite flakes, and finishing softening by friction with both hands.

Resulted use-wear

Stone tools used for the first tasks of fresh hide processing, like fleshing, present underdeveloped microscopic use-wear. It has been underlined that the work of fresh soft animal materials does not mark intensively artifacts edges. On the contrary, the stretching tasks, in particularly those involving abrasive substances, produced highly developed macro and microscopic wear (fig.12).
Figure 12 - Stone edges and surfaces of endscrapers made on quartz and quartzite before and after hide processing (200x): a, b, c) polishes produced by meat residues removal; d) edge rounding after scraping with ochre and ashes at 50x; e) edge rounding at 200x (Photos J.P.Ruas and micrographs M.de Araujo Igreja).
Experiments on other materials: fish, wood and bone

Other experiments are being undertaken. Results concern few tools used to filleting fish, scrape wood and engrave bone (fig.13).

![Fig.13 – a) filleting fish with quartz ; b) scraping wood with quartzite ; c) engraving bone with quartzite.](image)

**Resulted use-wear**

Some polish from scaling and filleting tasks were observed on quartzite (fig.14). They are not very developed, and this in spite of being used for more than 30 minutes.

![Figure 14 – Stone edges and surfaces before and after fish processing (200x): a) polish produced by scaling; b) polish produced by filleting at 100x ; c) polish produced by filleting at 200x.](image)
Concerning bone and wood, the use-wear developed is highly similar to that found on flint (fig.15).

Figure 15 - Stone edges and surfaces before and after bone and wood processing (200x) : a) polishes on quartzite produced by bone engraving ; b) wood scraping.

The archaeological application

The study of non flint stone tools from several Upper Palaeolithic sites located in north and central Portugal comprise in the research project, allows to confirming the effectiveness of the application of the proposed methodology (DIC + high resolution epoxy casts) for archaeological interpretation. First results presented here are issued from a post-doctoral project dealing with the wide range of technological and economical variability of human groups during the Upper Palaeolithic in Portugal. In Europe, Upper Palaeolithic corresponds to the spread of anatomically modern humans (35-10 thousand years BP), characterised by important social and technological developments. In Portugal, Upper Palaeolithic begins more recently that in the rest of Europe (around 30 000 years BP) and under specific environment conditions. Although the Portuguese sequence shares a number of general features with France and Cantabria, it presents some variation, in term of organisation of lithic and animal resources and in term of land-use (Zilhão, 1997). In terms of the lithic raw materials exploited, there is clear preference for non flint materials, like quartzite, quartz and rock crystal. Quartz reduction techniques and the blanks obtained are the same of those used on flint. Which seems to distinguish flint and quartz is that flint is preferentially used to obtain blades which are then transformed into tools as scrapers and burins, while quartz is mainly used to produce bladelets further used as projectile implements.
Given this «Lusitanian» context, particular from a climatic, biological and cultural point of view, the goal of the project is to characterise the relation between resource diversity and the organisation of technological systems on a global level, in terms of technological behavioural and resource exploitation strategies, by determining the activities, since acquisition to processing of natural resources (food quest and utilitarian needs) from archaeological sites functionally different: cave/shelter and open air located in Estremadura (Central Portugal) and in the famous Côa Valley.

By studying the kind of stone tools consumption trough use-wear analysis the goal is to best appreciate the specificities of natural constraints but also the cultural factors underlying the economic and social strategies in this territory.

The models available so far for Upper Palaeolithic stone tools consumption from specialised vs. residential occupations rely on archaeological assemblages mainly made of flint from Central and Eastern Europe and which paleoenvironmental conditions are different from Portuguese. Results obtained so far for the lithic industries of these sites are presented more extensively in another paper of the workshop proceedings. In broader lines, results show activities related with hunting (fig.16) and animal processing activities (fig.17).

Figure 16 – Projectile fractures on bladelets (50x) from the Gravettian site of Cardina (Foz Côa).
Use – wear is underdeveloped and few used zones per tool were observed independently of the site’s function. This testifies a tools consumption that is moderate, probably related with the easily accessible local raw materials and the absence of stress in their acquisition. Results contrast with those usually obtained for flint tools from distinct functional sites: Temporary occupations are associated with moderate use of stone tools and sedentary ones with highly used. This rises up the question of transferring the model of tools consumption, based on flint, into contexts which natural conditions and raw materials exploited are different.

Conclusions and perspectives

The present study has provided information regarding methodology in use-wear analysis of non flint stone materials. The key conclusions of this paper are summarised as follows.

Firstly, the association of the DIC microscope and epoxy casts proved to be highly efficient for the systematic functional analysis of non flint stone tool assemblages, independent of artifact color chemical weathering or length of objects. A further point observed was that some kinds of quartzite do not develop microwear. A comprehensive experimental program is being designed in
order to determine if this is related with the physical properties of these rocks and with their reaction to use-wear formation. Further insights will be gained on the extent of experiments to other kinds of quartz, quartzite and rock crystal, since these raw materials present a great variety.

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1 Scanning Electron Microscopy (Microscópio Electrónico).
2 Confocal Laser Scanning Microscope (Microscópio de Laser Confocal).