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THE COLOR OF TRANSFORMATION: INVESTIGATIONS INTO HEAT TREATMENT OF NATUFIAN ARTIFACTS FROM HAYONIM TERRACE (ISRAEL)

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ABSTRACT

In the Natufian lithic component at Hayonim, both in the cave and the terrace, numerous artifacts of pink/red color may be recognized. Cherts with similar appearance are not present in the geological environment surrounding the site in Northern Israel. Pink chert available in Jordan is shown to be of different nature. Thus this leaves us with the hypothesis of intentional heat treatment of locally available iron-rich yellow chert, of Cenomanian age. Based on experimental replication of chert firing and SEM analysis, we argue that a well-mastered and controlled use of fire was practiced by some skilled craftsmen at Hayonim throughtout the Late Epipalaeolithic.

KEYWORDS: Natufian, Near East, Lithic Technology, Heat Treatment, Pyrotechnology

1. INTRODUCTION

The site of Hayonim is located in Northern Israel on the Western slope of the Galilee, facing the Mediterranean Sea. It is composed of a cave and a terrace. The cave reveals a deep stratigraphic sequence, with numerous archaeological layers dated from the Mousterian period (250,000 BP) to the modern era (Bar-Yosef and Goren, 1973; Belfer-Cohen, 1988; Bar-Yosef et al., 2005; Mercier et al., 2007). The terrace yields a shorter occupation, extending from the Geometric Kebaran (ca. 15,000-13,000 uncal. BP) to the Pottery Neolithic (Henry and Leroi-Gourhan, 1976; Henry et al., 1981; Valla et al., 1989, 1991; Valla, 2012 ed.). Striking among all by the wealth and diversity of archaeological remains recovered is the Natufian layer (ca. 13,000-10,300 uncal. BP). One of these abundant categories of artifacts is represented by the lithic industry. This assemblage has traditionally been characterized by an overwhelming quantity of small irregular flakes, a small amount of short and wide bladelets, and small exhausted cores. The toolkit reveals usually a strong microlithic component, dominated by either backed bladelets or geometric microliths, especially in the shape of lunates (Bar-Yosef and Valla, 1979; Belfer-Cohen, 2002; Delage, 2011; Valla, 1984).

Research in the last two decades has made undeniable progress in our understanding of various aspects of the Natufian lithic production, notably typology, chronology and function. Yet, critical moments of the *chaîne opératoire*, such as the procurement and debitage, have been either unexplored or are relatively misunderstood. Recent efforts have been produced to correct this situation (Delage, 2001). The type of analysis and results presented in this paper are part of this new trend. The focus will be on a specific moment of the lithic reductional sequence, i.e. the use of fire for intentional heat treatment, that has not received much attention (but see Edwards, 1987; Edwards and Edwards, 1990; Delage and Sunseri, 2004).

We had in hand several artifacts of yellow and red color collected on the surface of a ploughed field on the lower terrace at Hayonim. Among them, two blades and a flake exhibited a reddish color and a fine-grained texture; two other blades and a core were of yellow color, and finally a blade exhibited a gradient of colors from one end to the other.

During the analysis of the Late Epipalaeolithic lithic assemblage at Hayonim Terrace, we noticed that both sets of colored cherts were present as small items; they also yielded similar fine-grained texture and cortex appearance (Delage, 2001, 2013). As a working hypothesis for the present research, we wondered whether there was a connection between these two sets; in other words, we questioned whether the yellow artifacts could be the unheated, raw counterparts of the red ones (characteristic of heat treatment?). Our ultimate goal was to provide evidence to support our argument that the red artifacts under consideration here were indeed intentionally heated.

2. LITHIC HEAT TREATMENT

The contact of stone artifacts with fire/heat can produce 1) items that are partially reddened at one extremity, leaving the rest of these objects with their original color; 2) items that reveal a darker color over their whole surface and show macroscopic stigmata easily recognizable (e.g. cracks, potlids); 3) pieces that are homogeneously reddish in color. Artifacts from the first two categories would be considered as, respectively, slightly and extensively "burnt". In other words, they are the result of an accidental action of heat/fire on them, which leaves little room for intentional intervention by human agents. Rather, such interpretive phrasing suggests significantly less control over the pyrotechnology at hand and the material characteristics effected in the lithics. Some objects of the third group could also be the product of an unintentional contact with fire/heat, but these would be extremely rare in any lithic assemblage. But, when reddish chipped stones are present in relative abundance in a lithic sample, we may have a strong presumption linking these items to heat treatment. In an effort to clarify the terminology currently in use, we should apply this specific expression only in the case of *intentional* thermal alteration (see Gregg and Grybush, 1976).

Definitions for the recognition of lithic heat treatment in the archaeological record have been offered since the 1960's, which have usually focused their attention on physical/mechanical changes/alterations, e.g. grain homogeneity and size, interstitial matrix structure and isotropy (Bordes, 1969; Purdy and Brooks, 1971; Collins, 1973; Hester and Collins, 1974; Flenniken and Garrison, 1975; Domanski and Webb, 1992; Domanski et al., 1994, 2009; Schmidt, 2013, 2014). These kinds of analysis often require the use of sophisticated analytical equipment and, since they are relatively expensive to run, concern a small sample of artifacts (Göksu et al., 1989; Griffiths et al., 1986; Melcher and Zimmerman, 1977; Pavlish and Sheppard, 1983; Richter et al., 1999, 2011; Robins et al., 1978; Schmidt et al., 2011, 2012, 2013; Toyoda et al., 1993; Valladas, 1983). From a different viewpoint, we offer here a minimal definition of heat treatment, emphasizing exclusively the macroscopic change in color. In this case, entire collections of lithic artifacts can be taken into consideration, giving better statistical reliability of the study (see also

Dowd, 1981; Lavin, 1983; Patterson, 1996; Cooper, 2002). The macroscopic criteria to define heat treatment we found most discriminant are twofold: 1) a reddish/pinkish color must affect the whole artifact in a homogeneous way (and this usually also goes deep in the siliceous matrix); 2) no features, such as fissures, potlids, etc. (which are clear evidence of overheating or burning) should be present.

Why then are these features the result of heat treatment? Intentional thermal alteration of siliceous rocks requires an understanding and mastery of the complex properties of combustion, temperature control, etc. (see Crabtree and Butler, 1964; Inizan et al., 1975-76; Inizan and Tixier, 2001; Purdy, 1978, 1982; Schmidt, 2014). In order to account for #2 (previous paragraph), any prehistoric flintknapper would have to know intuitively the mechanical/physical properties of the selected chert type when in contact with heat and at which temperature that specific rock crumbles and explodes. To account for #1, that same flintknapper would need to know the approximate location to set the artifacts in the hearth or pit, the temperatures involved, as well as the duration of the heating and cooling process for this pyrotechnological phase to be carried out successfully in a controlled and replicable manner, so that many lithic artifacts can yield similar macroscopic and mechanical/physical homogeneous transformations.

3. RED CHERTS IN THE NATUFIAN OF HAYONIM TERRACE (AND BEYOND)

Only a multi-dimensional reasoning and analytical procedure could sort out and clarify the problems involved here. In that sense, we first need to document that these red cherts are not present in sedimentary deposits located in Northern Israel.

As part of a techno-economic analysis of the lithic assemblage from Hayonim Terrace, we set as a goal to understand the procurement patterns and the organization of lithic production during the Late Natufian in that region (Delage, 2001, 2013). From the start, this study was carried out with a strong emphasis on lithic artifact sourcing. In order to determine their geographical origin, several methodological steps were followed: geoarchaeological survey to map and observe the chert-bearing geological deposits; collect of chert samples; establishment of a lithic reference collection ("lithotheca"); analysis (based exclusively on naked-eye visual observation) of these geological lithic samples; analysis of archaeological lithic artifacts using the same criteria and technique; comparison of the two sets of data to assess the source area of the archaeological material (Delage, 1997, 2001, 2007a, 2012, 2013).

Following this procedure, a total of 26 raw material types (predominantly chert) were identified in the archaeological assemblage. Most of these lithic types could be attributed to a specific geographical source area. Chert types of unidentified origin were present, for the most part, in extremely small amount. Thus, they did not appear to constitute a major (quantitative and statistical) obstacle to reconstructing the Natufian procurement patterns at Hayonim Terrace. These considerations would apply for most cherts of unknown origin, except for one. This unique type is, by contrast, much more abundant (ca. 15% of the total) than any other rock of undetermined provenance in this lithic assemblage. This type is characterized by siliceous rocks of a distinctive red color (Munsell Color Chart 10R 6/3-10R 3/4) (**Fig. 1a**). It is generally a very fine-grained material of opaque texture. When preserved, the cortex reveals a very fresh appearance, suggesting that these rocks were collected directly at the outcrop or at a close proximity. As similar examples, recent studies have documented artifacts of reddish color which were clearly the result of accidental firing at Manot Cave (Weiner et al., 2015, tabl. 1) and Neve David (Yeshurun et al., 2015, fig. 16, No 7). Since this chert type has not yet been identified in any geological deposit in the local environment around Hayonim Terrace and even beyond at the regional scale of Northern Israel, it poses a more serious problem to the picture we were trying to draw of the procurement patterns.

In the last two decades or so, red cherts have been a subject of investigation and controversy in the recent prehistory of Southwest Asia (see Delage and Sunseri, 2004). They were first identified in lithic assemblages dated to the Pre-Pottery Neolithic period and interpreted as intentionally heat-treated (Crowfoot-Payne, 1983, 629; Gopher, 1989; Miller, 1983; Nadel, 1988, 1989, 1997). Recently though, Leslie Quintero, and her colleagues Philip Wilke and Gary Rollefson, found chert nodules with pinkish color (**Fig. 1b**; see a description of this type further below) naturally occurring in Jordan, in Wadi Huweijir, near Amman and the nearby famous Neolithic site of Aïn Ghazal (Quintero, 1997, 1998). Pushing further their fieldwork, combined with an extensive review of the geo-archaeological literature, they realized that in situ sedimentary outcrops yielding red/pink cherts similar to those from Wadi Huweijir were in fact present at various locations in Jordan, and all seemed to be of Senonian (Campanian) age (i.e. Amman Silicified Limestone Formation) (Rollefson et al., 2007). "Huweijir-type cherts" are therefore a category of raw materials extremely important and meaningful for the reconstruction of prehistoric patterns of lithic procurement and exploitation in this region of Southwest Asia. Yet, as suggested by Nadel (1989, 1997), it is still possible that, in Neolithic lithic assemblages of the Southern Levant, pink/red cherts could combine both intentionally heat-treated cherts and non-heated cherts (that could now be assigned to the Huweijir type). Our own observations of the Pre-Pottery Neolithic lithic assemblage from C. Commenge's excavations at Munhata seem to support this assertion (Delage, 2007b).

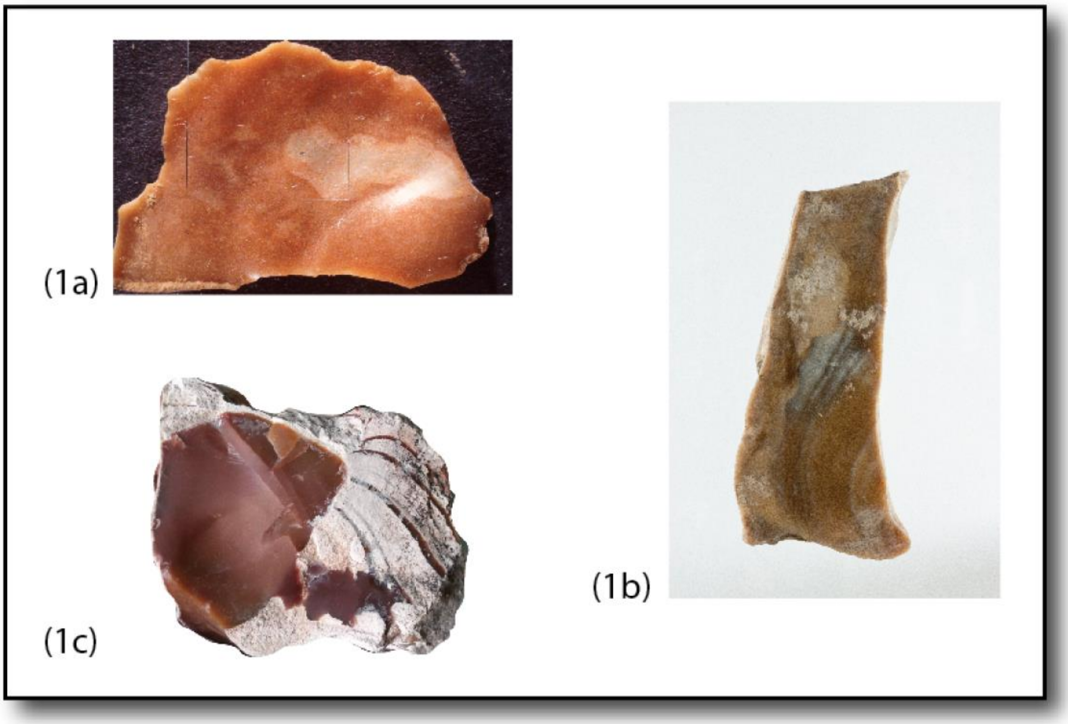

Figure 1. Various forms of reddish/pinkish cherts: 1a- Red chert, archaeological artifact from Hayonim Terrace (Israel), Late Natufian (photo: C. Delage); 1b- Huweijir-type chert, Amman Silicified Limestone Formation (Campanian), archaeological artifact from Wadi Hammeh 27 (Jordan), Early Natufian (photo: I. Capezio); 1c- Red chert, geological sample collected in Wadi Hammeh (Jordan), Amman Silicified Limestone Formation (Campanian) (photo: I. Capezio).

On another hand, recent work conducted in the Jordan Valley (as part of a renewed project at Wadi Hammeh 27) and further East, in the Azraq Basin (as part of the Kharaneh IV Project; Sanchez et al., 2014), has documented the presence in Late Cretaceous deposits (respectively in Amman Silicified Limestone Formation and Muwaqqar Chalk-Marl Formation) - and thus its widespread distribution in Jordan - of another type of red/pink chert, but this one is of intense homogenous color (**Fig. 1c**), often similar to that at Hayonim.

Based on these stimulating observations from both Neolithic lithic assemblages and chert types in Jordan, would it be possible then to assume that some sources yielding pink chert nodules be present in Northern Israel, but not yet identified? Senonian chert-bearing formations are indeed present in Israel, particularly in Galilee. It is important to note though that the Senonian stage has been further divided into several sub-stages (e.g. Santonian, Campanian, Maastrichtian), which do not hold the same potential in terms of chert-bearing sediments. In Northern Israel, but this is also the case for the whole country, the only geological unit of the Senonian to yield chert nodules is the Campanian, locally known as the Mishash Formation (Delage, 2007a). This sedimentary unit is particularly well documented in the Southern part (Negev) of the country, where the siliceous rocks represent massive deposits (e.g. Arkin et al., 1984; Kolodny, 1967, 1969, 1986; Kolodny et al., 2005; Steinitz, 1970, 1977). In Northern Israel, this unit is extremely rare, present in very thin and localized outcrops (Flexer, 1971; Kafri, 1972; Levy, 1983). Moreover, Mishash cherts (see Delage, 2001, Appendice 5) do not bear any visual resemblance with Huweijir-type cherts or any other pink chert from Jordan (see below). These various lines of arguments should convincingly demonstrate that pinkish cherts are not present in the regional geological setting of the Galilee.

We can now wonder whether the Natufian communities at Hayonim Terrace would have imported some pink chert from Jordan? To answer this question, we need first to look more precisely at the artifacts of pinkish color at Hayonim Terrace and compare them with Huweijir-type cherts. As described by Quintero and her colleagues (Rollefson et al., 2007), Huweijir chert reveals a highly siliceous matrix with a homogeneous, smooth and waxy texture. Moreover, even though its color is often characterized as pink and/or purple, this chert type shows "a range of color variation from place to place (and even within a single flint nodule)" (idem). In fact, besides the dominant shade of pink, other colors, such as yellow and light brown, are also present, in a subtle arrangement of colorful concentric bands (**Fig. 1b**). Therefore, this Huweijir chert is very specific and can easily be singled out by naked-eye visual examination. In sum, its macroscopic features are so distinctive that it cannot be mistaken with the artifacts at Hayonim and we can state with all certainty that it is not present in the Natufian component at this site.

By contrast, macroscopic observation often could not differentiate the reddish artifacts at Hayonim from the intense homogenous red/pink chert from Jordan. The only -- but extremely important -- difference would rest on the nature of the cortex. When exploited by prehistoric people, such as at Wadi Hammeh 27 (C. Delage, personal observations, dec. 2015), these pink cherts yield a very battered neocortex, characteristic of a secondary context of deposition, whereas at Hayonim reddish chert nodules reveal a red carbonate cortex witnessing a collect directly at or close to the outcrops. Thus it is strongly plausible that it was not imported from such a long distance, even though this hypothesis cannot be fully discarded at this stage; only a geochemical characterization of these two sets of rocks could resolve definitely this question.

The various geo-archaeological hypotheses put forward to account for the nature and origin of this reddish raw material in the lithic assemblage at Hayonim revealed themselves unfruitful. We then turned to an alternative scenario, which has motivated the research and evidence presented in this paper: could these reddish artifacts be the result of intentional heat treatment? In other words, we have just demonstrated that their unique color is not an original and natural feature, and we argue here that it could be a physical modification due to thermal action. Moreover, since there is no evidence of the usual stigmata associated with the accidental contact of siliceous rocks with fire (e.g. cracks, potlids, etc.), we would consider this process as a well-mastered pyrotechnology (Delage and Sunseri, 2004).

Such a working hypothesis does not appear too unreasonable in the Late Epipalaeolithic context of the Levant, since similar behaviors have been documented elsewhere. At most sites dated to this period, large amounts of burnt lithic fragments are noticeable by their characteristic features: cracks, potlids, etc. Nevertheless, lithic artifacts are also present which reveal patterns of more intentional and controlled use of heat/fire. In these cases, the specimens concerned show usually a reddish color, associated with a very fine texture and a shiny/glossy surface. Based on the available literature and personal observations, several sites have been mentioned revealing such patterns: Wadi Judayid/J2 (Sellars, 1989, 42-43; Henry, 1995, 326), Wadi Hammeh 27 (Edwards, 1987, 196-204; Edwards and Edwards, 1990), Wadi Hisban 6 (Edwards et al., 1999, 43), Nahal Sekher 23 (Goring-Morris et al., 1998, 165; Marder, 2002, 189), Azariq XV (Goring-Morris et al., 1998, 165; Marder, 2002, 189), Shunera VII (Marder, 2002, 189), Hayonim Cave and Terrace (Delage, 2001), and 'Eynan (Delage, 2001).

4. METHODOLOGICAL CONSIDERATIONS

Throughout the abundant literature, there appears to be several ways implemented by scholars to characterize heat-treated lithic artifacts. One of them requires the use of techniques such as Thermoluminescence (TL) or Electron Spin Resonance (ESR) (Göksu et al., 1989; Griffiths et al., 1986; Melcher and Zimmerman, 1977; Pavlish and Sheppard, 1983; Richter et al., 1999, 2011; Robins et al., 1978; Toyoda et al., 1993; Valladas, 1983). Another procedure involves the re-heating of archaeological artifacts. Scholars, such as D. Nadel, or L. Quintero and P. Wilke, implemented this protocol (Nadel, 1988, 1989; Quintero, 1997, 1998). Experimental heat-treatment is yet another path of investigation. Following this latter approach, we developed a methodology where heating experiments played a major part. We also chose to complement this study with the a priori safe and useful technique of Scanning Electron Microscopy, since it is the most commonly applied approach to characterize lithic heat treatment. Our items of yellow cherts were thus selected for various destructive testing.

Before using this set of yellow artifacts for heating, we needed to address another logical step of our reasoning: ascertain the geological relationship of the red cherts with the yellow ones. In other words, we needed to prove that both sets of archaeological lithics under consi-deration (red and yellow cherts) were of the same geological formation and age, if not of the same source area. As a first step in that direction, the comparison of visual traits turned out quite conclusive, but it was not sufficient nor enough. We then performed a geochemical analysis on both sets of artifacts, as part of a larger geo-archaeological project to determine the sources of cherts used by prehistoric peoples in Northern Israel (Nathan et al., 1999). Results were more probant: they confirmed that both sets were of the Deir Hanna Formation (Upper Cenomanian), based on their signature in trace/rare earth elements. Thus, both the red and yellow cherts may be related to geological type Z08 (see Delage, 2001, append. 5). These Z08 type cherts are present in numerous localities in Western/Central Galilee, notably in major valleys around Hayonim: Nahal Meged, Nahal Sha'al, Nahal Ga'aton, Nahal Yehiam, Nahal Keziv, Nahal Hilazon, etc. (see Delage, 2001, fig. A5.49). In a recent study on Neve David (Mount Carmel), some lithic artifacts of similar yellow color and Upper Cenomanian age (Shamir Formation) yield a reddish tip proving the transformation of color from yellow to red related to the unintentional -- in this case -- effect of fire on these lithologies (Yeshurun et al., 2015, fig. 16, No 4-5).

In contrast, in a recent analysis trying to document possible lithic heat treatment at Manot Cave (Israel), the research team selected local cherts of the Sakhnin Formation (Upper Cenomanian) as the experimental referential material. They did not provide though any description (color, texture) of these geological samples. Yet it is now known that cherts attributed to the Sakhnin Fm., especially in Central Upper Galilee, around Nahal Keziv- Nahal Sha'al, are embedded in a hard dolomitic sediment and are made of a coarse-grained texture (Delage, 2001, vol. 2, 178-180, 219-221, fig. A4.7). We wonder if local Deir Hanna cherts would not be a better candidate for lithic procurement and exploitation at Manot Cave.

Prior to heat treatment, each artifact considered in this analysis was recorded, weighed and measured. The surface orientation and location of specific scars were carefully observed on each flake for magnified imaging. The best imaging site was marked and photographed for identification records. Samples were then placed in ceramic crucibles, on top of 3 cm of sand buffer and then covered with three additional cm of sand buffer, maintaining at least 3 cm of space between the sample and the crucible walls. This buffer prevented damage to the kiln wall bricks in the event of a catastrophic failure of the chert pieces during heating. The heating process was done in a Skutt KM 1027 automatic kiln, fitted with an Envirovent system. While the heating regime of previous workers (Purdy, 1974; Domanski and Webb, 1992) was originally targeted for the sample population, total structural failure and crumbling of material was observed after removing samples that had been heated to 500ºC. Temperature profiles at 200°C, 300°C, 400°C and 500ºC were therefore established. All samples were placed in the kiln in pairs of small ("tst" series) and large ("Btst" series), in separate crucibles, buffered similarly with sand. The one pair each was then heated using a ramp/hold firing of the kiln, to 200, 300, 400, and 500ºC respectively as per the heating experiments of Crabtree (and Butler, 1964) and following other investigators (Purdy, 1971; Rowney and White, 1984; Domanski et al., 1994). The kiln raised internal temperature by 100ºC per hour, maintained the peak temperature for 2 hours, and then cooled naturally. After heat treatment, samples were removed from their sand buffers and pressure flaked along a fracture surface defined at the preheating state and identified by mapping dots. This was done in order to maintain any possible expression of grain along that plane and to compare the oft-observed change in luster along a freshly flaked surface to the original surface texture. The artifacts were then visually observed and photographed for changes in color, lustre, etc.

In the next stage, the specimens were each mounted to a SEM stub of epoxy resin. They were then etched with hydrofluoric (HF) acid; and finally, ultrasonic cleansing to remove reaction agents and deposits was followed by a drying phase. After that, they were coated with a thin carbon layer, before examination under SEM after cross-referencing with archived test ID images to relocate and confirm image area mapping dots. A new set of images was then taken.

5. EXPERIMENTAL HEAT TREATMENT

The single core of unheated yellow chert from the Deir Hanna Formation was flaked into eight samples: four ("test1" through "test4") were 2 cm or less in length with mean mass of 0.5 g, four ("Btest1" through "Btest4") were 3.5 cm or greater in length with mean masses of 2.5 g (**Table 1**). The samples were separated into two series of test sizes ("tst" and "Btst") in order to bring representative samples of differing heat capacities, smaller mass and larger mass, to the hypothesized heat treatment conditions of 300 to 600ºC (see Purdy, 1974). As will be discussed below, it was discovered that this lithology cannot survive temperatures approaching 500ºC without structural failure, so the heating regimen for the final pair ("tst4" and "Btst4") was heated to 200ºC, rather than 600ºC, to examine the lower end of results from a contiguous heating spectra.

In addition, two geological samples of Monterey Banded Chert (California) were also subjected to the same analytical procedure for reference and comparison. They were selected for both a low-angle arris as well as a high-angle arris. Additionally, they were mounted and mapped for dorsal and ventral surface representations. During the development of the mounting, etching, and heat treatment procedures, these two samples were utilized before any archaeological or experimental samples were introduced to minimize exposure of those samples to potentially damaging mistakes and test methods.

Table 1: Yellow chert exposed to heat showing various color changes with temperatures.

Temperature	Pre-heated color	Post-heated color	Fracture changes
200º C	10 YR 7/3	10 YR 6/4	No
300º C	10 YR 7/3	5 YR 4/4	No
400º C	10 YR 7/3	5 YR 3/3	No
500º C	10 YR 7/3	5 YR 5/2	Yes
600º C	10 YR 7/3	5 YR 2.5/1	Yes, very friable

It was observed (**Table 1**) that the test pieces heated to 300ºC were closest in color (light brown to red) to the "red" archaeological samples (dark red to brown). However, the slight red coloration of the test samples heated to 200ºC suggests that the artifacts from Deir Hanna formation were most likely heated to a temperature somewhere between 200 and 300ºC.

The most dramatic change in material properties occurred at temperatures above 400ºC, which was commensurate with a change in color to dark grey. Above this temperature, the chert often became friable and no longer retained the structural properties for successful flaking. In some extreme cases, where samples were heated to 600ºC, they fractured and fell apart inside their sand buffer. Samples which obtained this stage of heat treatment were the only samples to exhibit clear alteration of fracture properties from pre- to post-treatment imaging. However, their fragility in actual flaking makes it highly unlikely that they would be utilized as formal tools. Additionally, these samples were the farthest from the color spectra of the archaeologically observed chert artifacts. We suspect that several artifacts from Manot Cave which yield black/dark grey color, as well as potlids and cracks (Weiner et al., 2015, table 1), were exposed to similar high temperatures.

Finally, the large amount of iron in both heated and raw cherts of the Deir Hanna Formation (type Z08) plays an essential contribution to color variation from intense yellow in unheated specimens to the oxidation of Fe toward reddish colors in chert samples exposed to heat.

In sum, the specific red color of this chert type (reflecting a high content in iron oxide) would be the result of intentional heat treatment.

6. SEM ANALYSIS

Images captured during electron microscopy revealed lepispheres within the silicate matrix of the chert. These "bump" structures exhibited preferential fracture propagation around their outer boundaries and were further exposed after HF acid etching. Acid overetching after heat treatment may have obscured these structures beyond recognition in post-pressure flaking photomicrography.

Barite crystals, rhombic in shape, are observable throughout the lithology. Fracture wave fronts propagate preferentially around them before and often after heat treatment. However, a few examples of the crack propagation through barite crystals were observed (**Fig. 2**). This suggests that heat treatment may be altering the mechanical properties of the crystals in somewhat the same way as the larger substrate.

Most interestingly, the directions and extent to which fracture wave fronts traveled around and through the barite crystals, especially in relation to freshly-flaked heat treated samples, differed depending on the relative location of the new flake on the original fracture surface (**Figures 2, 3**). It appears that debitage activities after heat treatment have a different effect on grain expression when the force applied is along the plane of the original flake scar. A greater sample size and a satisfactory means of dealing with charge accumulation is needed before this can be better investigated.

Magnetization of the samples after flaking increased the amount of charge per unit accumulating on the sample surfaces. This dramatically reduced the window of opportunity for looking at the surface in the SEM and may only be remedied after complete gold sputter coating to carry off the charge. However, some images of the fracture surfaces after heat treatment were observable and captured for comparison to the unaltered/heated surface textures. The resultant "sub flaking" (**Fig. 2**) and continued fracture propagation around intrusive barite crystals was inconclusive and the accumulation of charge was too severe to image larger or more strategically placed locations. More time is needed to deal with the charge problem, capture a larger sample of comparative images, and interpret the surface morphology at those magnifications.

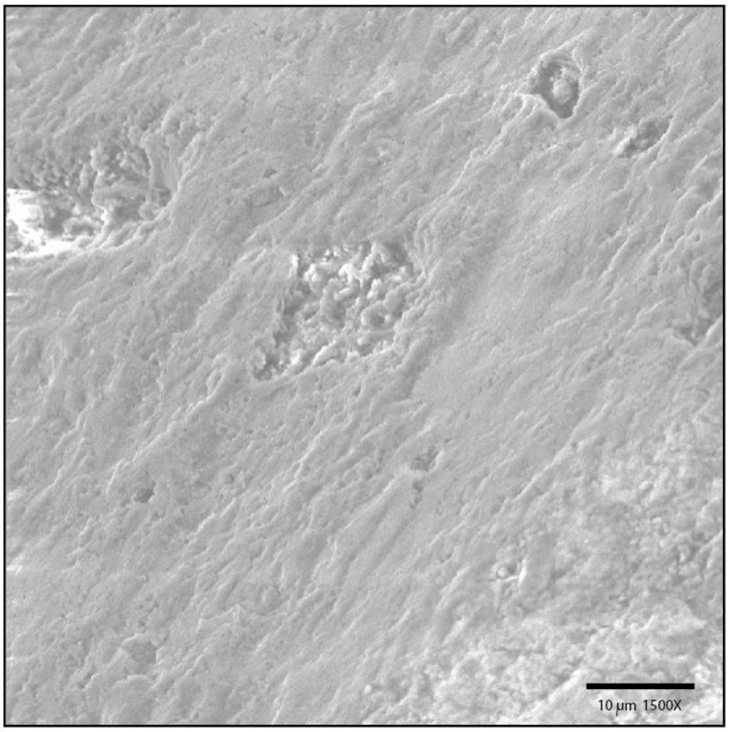

Figure 2. Fracture propagation through barite crystals of sample heated at temperatures twice as high as archaeological specimens.

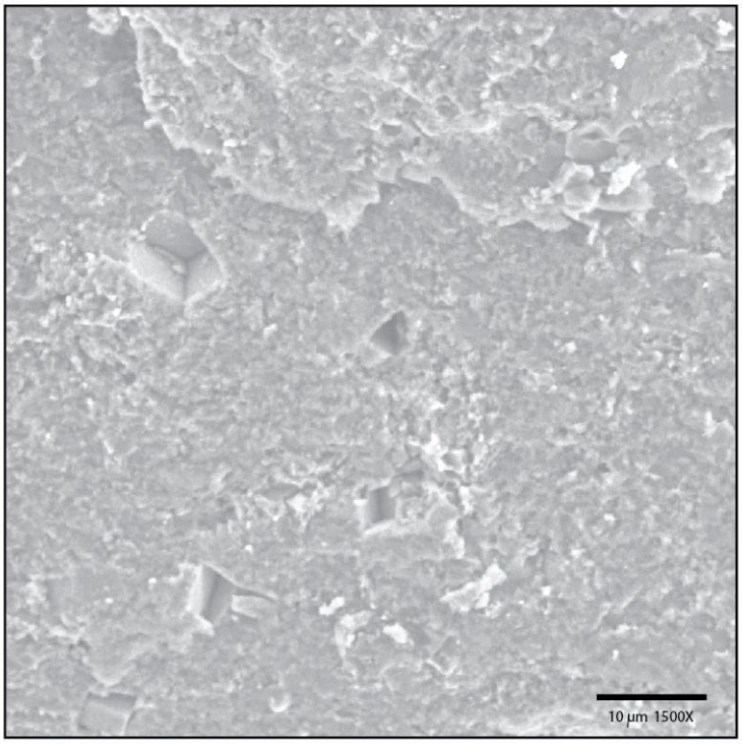

Figure 3. Fracture propagation around barite crystals along a pressure flaked scar after heat treatment.

7. CONCLUSION

One of the conclusions that our research led us to sheds light on the obvious contradiction between experimental heat treatment and the actual archaeological facts of prehistoric pyrotechnological behaviors. Indeed there is a serious methodological concern regarding the temperature used in previous experimental analyses and its relevance to document archaeological lithic heat treatment. Most of the experiments published in the literature have generally used temperatures above 300ºC in their heat treatment protocols (Domanski and Webb, 1992; Draper and Flenniken, 1984; Mandeville, 1973; Purdy, 1974; Schmidt and Fröhlich, 2011; Schmidt et al., 2011, 2012; Wadley and Prinsloo, 2014). Yet, even though cases of rocks (e.g. quartzite, silcrete, etc.) intentionally heated at high temperatures (ca. 400-500ºC) may be documented in the archaeological record, they are usually rare. It appears in fact that most cases range between 200-350ºC (e.g. Schmidt et al., 2013). Based on these lines of evidence, we may wonder about the theoretical justification for such methodological procedures when they only fit the evidence from the archaeological record in some rare occurrences.

In light of this proposition, the Late Epipalaeolithic/Natufian context does not appear any different. The study carried on lithic artifacts from Wadi Hammeh 27 by Ian and Phillip Edwards was the first to document such a pattern (Edwards and Edwards, 1990, 3). Similarly, in the heating experiments related to Natufian artifacts reported in this paper, it is noted that the closest correlations to colors observed in the archaeological assemblage were recreated in the 200º to 300ºC range. By contrast, temperatures ca. 500ºC caused extensive damage to the integrity of Cenomanian chert.

This result leads to some other closely related conclusions. Researchers have noticed that higher temperatures are needed to achieve mechanically desirable changes in the structure of heat treated lithics and that these changes, visually observable on artifacts, are progressive - but profound and irreversible: the first stage is color modification toward reddish shades; luster is then appearing; etc.; at the final stages, a change to dark colors, associated with fissures and potlids, may be observable.

In the archaeological case of Hayonim Terrace presented here, cherts of the red type may reveal a noticeable color change, induced by temperatures which were though not effective and high enough to produce luster change or any further modification. Similarly, SEM analysis on Wadi Hammeh 27 artifacts showed no major structural differences between raw and cooked cherts: "The surfaces of all the unheated Jabal Sartaba samples have a convoluted topography composed of bumpy granular aggregates. This pattern does not change significantly in the Jabal Sartaba samples treated at 250ºC and 300ºC" (Edwards and Edwards, 1990, 3).

Thus the traditional use - in the literature - of the luster criteria to identify when heat treatment took place in the reductional sequence would not work in some cultural contexts, such as the Natufian. Indeed looking at the flaking scars on flakes or cores to associate mat and glossy surfaces with - respectively - pre- and post-heating is totally irrelevant in such a context. Yet, the presence of all technological classes of artifacts (i.e. chunks, debitage, cores, and tools) in reddish chert suggests that the controlled use of fire was implemented at an early stage of the *chaîne opératoire*, before the actual flaking and blank production took place.

Furthermore, if prehistoric craftsmen applied temperatures ca. 200-350ºC, as documented in most prehistoric cases, it would be quite impossible to clearly identify any changes in physical or mechanical properties using SEM analysis. This technique does not appear indeed to be an appropriate methodological procedure in documenting intentional heat treatment in most archaeological cases. In any project of heat treatment characterization, a stage for assessing the temperature involved (using TL, ESR, or experimental observation) should take place in the analytical procedure before any attempt at applying SEM analysis. In most cases, SEM won't be relevant, even though a review of the specialized literature shows that it is the most commonly used technique for intentional heat treatment characterization.

By contrast, it would be interesting to further investigate the relationships between temperature and structural changes. Overall, the type of rock considered (chert, quartzite, silcrete, etc.), the nature of mineral composition (quartz, opal-A, opal-CT), etc. might be major components to take into account in the explanation of changes occurring with heat treatment. We suggest some possible answers here. An experimental methodology, involving different angles of blow, is to be used in conjunction with a microstructural theoretical model, to reveal the extent of "grain" orientation change with heat treatment. Similarly, an investigation of the vector components of fracture propagation would address relations of isotropic material response. These can dovetail neatly into a program that uses fracture toughness tests to create a response profile for a lithology. Refinement of models of lithic microstructure may allow archaeologists to explain not only changes that result from heat treatment, but also a host of related lithic responses to human use including use-wear, knapping material fracture preferentiality, and impact on reduction strategies. A most complimentary methodology to this study could be the characterization of grain size via X-ray diffraction methods. Spikes in the 212 angstrom peaks for certain elements have been correlated to grain size within the sample material (see Domanski and Webb, 1992). This would provide a quantitative measurement for the changes observed under SEM and allow the investigation to draw more definitive conclusions from research across lithologies, providing a multi-regional analysis tool.

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