

## THE EARLIEST USE OF CORUNDUM AND DIAMOND, IN PREHISTORIC CHINA\*

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*The majority of prehistoric lithic artefacts were fashioned from rocks and minerals no harder than quartz, and there is no prehistoric evidence for the working of harder materials, such as corundum and diamond. The earliest physical evidence for the use of corundum (ruby, sapphire) is thought to be the abrasive grit recovered from Bronze Age Minoan quartz beads (c. 1700–1500 BC), while diamond is thought to have been used no earlier than 500 BC, in India. Here we show that corundum was worked c. 4000–3500 BC during the Neolithic period in China, in the form of polished axes from the Liangzhu and Sanxingcun cultures. We also present physical evidence that later Liangzhu axes (c. 2500 BC), made from the same previously undescribed rock whose most abundant component is corundum, were polished to a mirror-like finish with a diamond abrasive. Our findings, which are the first to support the use of corundum and diamond in a prehistoric context, may also help to explain the trademark feature of the Neolithic in China, vast quantities of finely polished nephrite jade artefacts.*

**KEYWORDS:** CORUNDUM, DIAMOND, POLISHING, ANCIENT CHINA, NEOLITHIC, ELECTRON MICROPROBE, ATOMIC FORCE MICROSCOPY, REPLICATION EXPERIMENT, LIANGZHU, SANXINGCUN

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## INTRODUCTION

The overwhelming majority of lithic artefacts recovered from prehistoric archaeological contexts were fashioned from minerals, or rocks containing minerals, no harder than quartz (7 on Moh's scale). In China, the most notable are the highly polished and lavishly decorated ritual objects of nephrite jade (Moh's 6.5; see Rawson 1995). Diamond (Moh's 10) and corundum (Moh's 9), the hardest and second-hardest naturally occurring minerals (Klein and Hurlbut 1985), have been thought to be completely absent among prehistoric artefacts (Lucas and Harris 1962; Moorey 1994). In this report, we analyse three axes from the Liangzhu culture (Rawson 1995) and the recently discovered Sanxingcun culture (Joint Archaeological Team at Sanxingcun 2004) along the Yangzi river in southern China, dating from the beginning of the fourth millennium BC. These axes are contemporaneous with the earliest Neolithic worked stone tools from the lower Yangzi river valley, and their most abundant component is corundum. We also analyse a later Liangzhu axe from the third millennium BC, and via a replication experiment demonstrate that its mirror-like surface polish was most likely produced using a diamond abrasive.

## EXPERIMENTAL METHODS AND RESULTS

*Sample characterization, microscopy and microanalysis*

We used several techniques to characterize axes from tombs 41 and 273 (*c.* 4000–3800 BC, Figs 1 (a) and 1 (b)) of the Sanxingcun culture at Jiangsu Jintan Sanxingcun [SXC] (Joint Archaeological Team at Sanxingcun 2004); from tomb 124 (*c.* 3500 BC, Fig. 1 (c)) of the Liangzhu culture at Jiangsu Nanjing Beiyinyangying [BYYY] (Nanjing Museum 1993); and a highly polished axe fragment (*c.* 2500 BC, Fig. 3 (a)), a surface find from the Liangzhu culture site at Zhejiang Yuhang Wujiabu [YW] (Wang 1993). Using powder X-ray diffraction and electron microprobe (Cameca MBX, Noran TN-5502 energy-dispersive spectrometer (EDS), stage automation) modal analysis of about a thousand points in a square grid covering each sample (approx. 2 mm<sup>2</sup> total area), we find that all four axes are composed of corundum (40%), diaspore (33%) and muscovite (25%). All samples had the minor constituents rutile, pyrite, zircon and monazite (containing Ce, La, Nd and Pr), verified by EDS in the scanning electron microscope (SEM). Compositions from the microprobe are reported in Table 1. The corundum is in the form of micron-scale grains, whose crystallographic orientations are randomly distributed in space, surrounded by regions of diaspore and muscovite. Images were collected using the SEM (Fig. 2), where a combination of EDS, imaging with backscattered electrons and mapping electron backscattered diffraction patterns allows determination of the chemistry, crystal structure and orientation of the same crystal grains. All four samples, covering three sites and spanning more than a millennium, appear to have been fashioned from the same particular type of rock.

The YW axe fragment belongs to a class of axes dating to *c.* 2500 BC, all fashioned from a mottled brown and grey stone and polished to a mirror-like finish (Fig. 3). As a surface find, it lacks an excavated sample's unassailable provenance. However, its composition matches the three excavated samples that we have characterized, and its glossy surface is macroscopically identical to well-provenanced axes excavated from tomb 8 at Jiangsu Jiangyin Gaochengdun [GDC] (Joint Archaeological Team at Gaochengdun Site 2001; Nanjing Museum 2001) and tomb 9 (Figs 1 (d) and 3 (b)) at Shanghai Qingpu Fuquanshan [FQS] (Huang 2000), in addition to



Figure 1 Excavated Neolithic axes (author's photographs). (a) SXC M41:5 from Jiangsu Jintan Sanxingcun (c. 4000–3800 BC), length 13.18 cm. (b) SXC M273:3 from Sanxingcun (c. 4000–3800 BC), length 13.67 mm. (c) BYYY M124:3 from Jiangsu Nanjing Beiyinyangying phase II (c. 3500 BC), length ~15 cm; cf., Nanjing Museum (1993), pl. VI.2. (d) FQS M9:18 from Shanghai Qingpu Fuquanshan (c. 2500 BC), length 22 cm; cf. Huang (2000), pl. IX'.

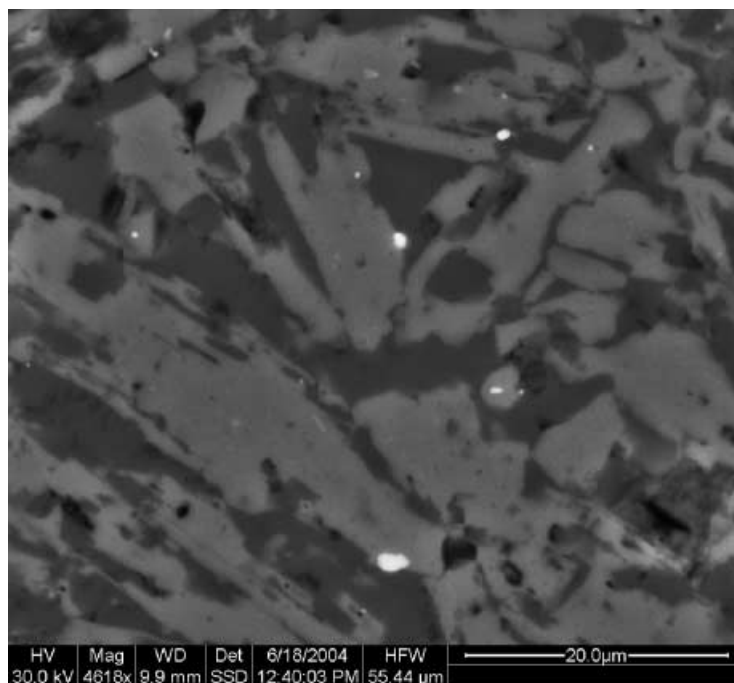


Figure 2 The surface of axe SXC M41:5 from Jiangsu Jintan Sanxingcun (c. 4000–3800 BC), imaged with backscattered electrons in an SEM. Lighter grey, angular corundum grains, ranging in size from several microns to several tens of microns, are surrounded by a darker diaspore. The small bright white, micron-sized crystals are rutile.

Table 1 Electron microprobe modal analysis of corundum axes

|                       | SXC M41:5<br>(c. 4000–3800 BC) | SXC M273:3<br>(c. 4000–3800 BC) | BYYY M124:3<br>(c. 3500 BC) | YW<br>(c. 2500 BC) |
|-----------------------|--------------------------------|---------------------------------|-----------------------------|--------------------|
|                       | <i>Percentage of phase</i>     |                                 |                             |                    |
| Corundum              | 39.0 ± 1.9%                    | 35.1 ± 1.9%                     | 39.1 ± 1.8%                 | 41.7 ± 2.1%        |
| Diaspore              | 36.6 ± 1.8%                    | 38.6 ± 2.0%                     | 28.0 ± 1.5%                 | 24.8 ± 1.6%        |
| Muscovite             | 19.8 ± 1.4%                    | 19.9 ± 1.4%                     | 28.9 ± 1.5%                 | 23.8 ± 1.6%        |
| Chlorite              | 0.0 ± 0.0%                     | 0.0 ± 0.0%                      | 0.0 ± 0.0%                  | 1.2 ± 0.3%         |
| Kaolinite             | 0.2 ± 0.1%                     | 0.0 ± 0.0%                      | 0.0 ± 0.0%                  | 0.0 ± 0.0%         |
| Zircon                | 0.0 ± 0.0%                     | 0.3 ± 0.2%                      | 0.2 ± 0.1%                  | 0.2 ± 0.1%         |
| Apatite               | 0.5 ± 0.2%                     | 0.1 ± 0.1%                      | 0.4 ± 0.2%                  | 0.0 ± 0.0%         |
| Monazite              | 0.2 ± 0.1%                     | 0.0 ± 0.0%                      | 0.0 ± 0.0%                  | 0.0 ± 0.0%         |
| Calcite               | 0.0 ± 0.0%                     | 0.0 ± 0.0%                      | 0.0 ± 0.0%                  | 0.2 ± 0.1%         |
| Rutile                | 2.9 ± 0.5%                     | 3.5 ± 0.6%                      | 3.4 ± 0.5%                  | 3.5 ± 0.6%         |
| Pyrite                | 0.8 ± 0.3%                     | 2.5 ± 0.5%                      | 0.1 ± 0.1%                  | 4.5 ± 0.7%         |
| Total points analysed | 1085                           | 987                             | 1205                        | 991                |



Figure 3 Liangzhu culture corundum axes reflecting patterns of squares (author's photographs). (a) YW fragment (c. 2500 bc); the square edge length is 1.0 mm. (b) Detail of FQS M9:18 (c. 2500 bc; Fig. 1 (d)); the square edge length is 20.0 mm. (c) F1998.4 from the Freer Gallery of Art, Washington, DC; the square edge length is 20.0 mm.

an unprovenanced axe F1998.4 in the Freer Gallery of Art (So 1998; and see Fig. 3 (c)), whose composition we found to match the others in this study. Macroscopically, all of the axes bear the same remarkable surface polish and will reflect an image like a mirror (Fig. 3). The YW fragment is particularly advantageous to the present study, because we could destructively sample a far larger portion of the intact surface (approx. 1 cm<sup>2</sup>) than would have been possible from the complete axes above (as historical artefacts and highly valuable works of art, they cannot be sampled to such a large degree), allowing quantitative characterization in an atomic force microscope (AFM), where intact axes were far too large to fit inside, and the replication experiment described below. Using the AFM, we found that the microscopic corundum grains, which give the YW fragment's surface its high reflectivity, have a root-mean-square roughness of only a few *nanometres*.

### *The replication experiment*

How these axes were polished to such a high degree is not known. Although the physics of polishing is poorly understood (Lu 1999), a harder abrasive will probably better level an initially rough surface. Corundum has twice the Knoop indentation hardness as quartz (Lide 1998), and given its abundance in these highly polished axes, their production may have required an even harder abrasive. The only natural material harder than corundum is diamond (Klein and Hurlbut 1985).

As no vestigial abrasive was found on the polished surface of the YW fragment, we performed a replication experiment to determine the identity of the abrasive used. Slices were taken from the inside and polished with the best commercially available forms of several different abrasives (quartz, corundum and diamond), and then compared using atomic force microscopy to the original surface polished by Liangzhu craftsmen in antiquity. Using a diamond saw, we cut three slices of rock from inside the YW fragment parallel to an original polished face of the axe, and embedded them in epoxy. We then ground the three samples in a Buehler Automet/Ecomet automated polishing machine using Buehler consumables. In all cases the sample holder rotated at 60 rev min<sup>-1</sup> in a direction opposite to that of the polishing wheel, so only the wheel velocity is subsequently noted. The three samples were first ground in a 9 µm diamond/oil suspension against a Hercules-H lapping pad under 15 lb of pressure at 120 rev min<sup>-1</sup> for 3 min, and then with a 3 µm diamond paste on an Ultrapol cloth at 15 lb and 240 rev min<sup>-1</sup> for 3 min. Each sample was then polished in a sequence of steps, all at 15 lb of pressure and 120 rev min<sup>-1</sup>, using the Texmet 1000 polishing pad, but each with a different abrasive.

We selected the commercially available abrasives that best approximated the natural diamond (natural diamond in a chemically unreactive oil suspension) and corundum (neutral-pH synthetic alpha-alumina suspension) potentially available to Liangzhu lapidaries. And for comparison, we also used the best modern quartz-based abrasive, a colloid of 60 nm silica particles in a high-pH solution that acts via a chemo-mechanical planarization (CMP) process; it is used to polish silicon wafers for microelectronics applications. The diamond-polished sample was polished with 1 µm diamond paste for 9 min, then with 250 nm diamond paste for 12 min and finally in a 100 nm diamond-oil suspension for 15 min. The alumina-polished sample was polished with 1 µm Micropolish alumina-water suspension for 3 min, then 300 nm Micropolish alumina suspension for 3 min and finally with 50 nm MasterPrep alumina suspension for 12 min. The silica-polished sample was polished with 60 nm MasterMet 2 colloidal silica for 30 min. Because the abrasive concentration varied, the total polishing times were not the same.

Each sample was polished with a given abrasive size until further polishing did not improve the surface; that is, scratches left by the next larger abrasive had been polished away completely, and the surface no longer changed with further polishing, as determined by optical microscopy. At this point, the sample was cleaned thoroughly and then polished with the next smaller abrasive size, using a new pad; the cleaning is required to prevent scratching from left-over larger abrasive particles. The cycle was repeated up to the smallest abrasive size listed above. In this way, we achieved the best possible polish with each abrasive.

#### *Atomic force microscopy and data analysis*

To gather quantitative height data, the surfaces of the YW axe were mapped with a Digital Instruments Dimension 3100 atomic force microscope using Olympus Tapping Mode tips. Data were collected in Tapping Mode for 100  $\mu\text{m}$  by 100  $\mu\text{m}$  areas of the surface (512  $\times$  512 data points) at a scan rate of 0.2 Hz. A first-order tilt correction was applied to the data. AFM maps of square areas on the three polished surfaces and the surface created in antiquity are displayed in Figure 4.

Qualitatively, of the three polished samples, the diamond-polished surface most closely matches the surface polished in antiquity. They both are flat, smooth and occasionally interrupted by sharply defined pits a few micrometres in length (Figs 4 (a) and 4 (b)). The edges of the corundum grains are not accentuated, and the heights of adjacent grains generally differ by

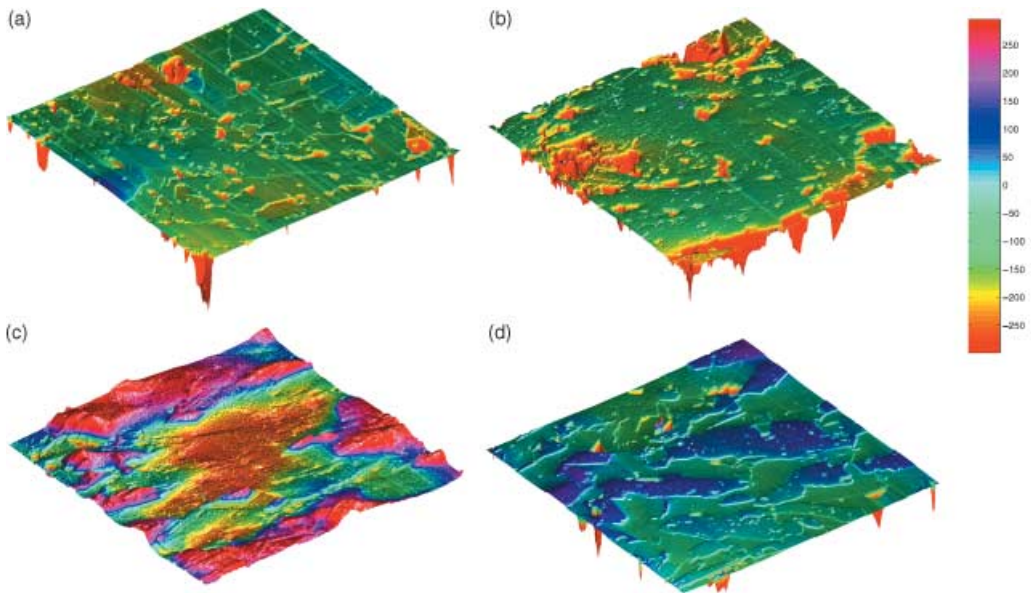


Figure 4 Atomic force microscope (AFM) data from polished sections of the YW axe fragment. The images are of 100  $\mu\text{m}$  by 100  $\mu\text{m}$  areas on the sample surfaces; the tick marks on the z-axis height scale correspond to 50 nm intervals. (a) The surface of the YW fragment polished in antiquity. (b) A diamond-polished section. Orange pits in (a) and (b) denote areas where grains have pulled out in the course of polishing. (c) An alumina-polished section, with very rough gouging. (d) A silica-polished section, showing rounded corundum (blue) hills over a lowered diaspore (green) ground.

only 10–20 nm. By contrast, the alumina-polished sample shows much more gouging and roughness (Fig. 4 (c)), with steep topography and deep pits, tens of microns across, distributed over the entire surface. Likewise, polishing with colloidal silica also fails to reproduce the surface polished in antiquity: individual corundum grains appear as pronounced round bulges over a lowered diaspore ground (Fig. 4 (d)).

Two quantitative tests also identify the diamond-polished surface as best matching the surface polished in antiquity. Height histograms (centred around the median height) of the four samples are given in Figure 5 (a). Histograms of the diamond-polished surface and the surface polished in antiquity have nearly the same width and similar shapes. By contrast, the alumina-polished surface has a far lower and broader distribution; the silica-polished surface is both wider and bimodal, with a second peak to the right of the main one arising from the corundum bulges. The second quantitative metric, normalized azimuthally averaged height–height correlation functions of the form:

$$H(r) = \langle |h(r) - h(0)|^2 \rangle / h(0)^2 \quad (1)$$

are plotted for each sample in Figure 5 (b), showing how the roughness of each surface changes over different length scales. The diamond-polished surface and the surface polished in antiquity have very similar pits extending several micrometres below the surface, created when smaller grains are pulled out in the polishing process. These pits do not reflect polishing technique, but rather local grain boundaries, and are steep enough that the sides of the AFM tip bumps against the walls. We therefore calculated the correlations in Figure 5 (b) after excluding points at a depth greater than 300 nm below the median (i.e., zero depth in Fig. 5 (a)) for all samples; the results were insensitive to moving the cut-off by  $\pm 50$  nm.

A rough polished surface may be imagined as a surface with quenched disorder, such as that generated by thermodynamic capillary waves (Sachdev and Nelson 1984); in such systems,  $H(r)$  grows as  $\log(r)$  (Weeks 1980). To test this, each of the four curves was fitted to a function of the form:

$$H(r) = \beta[(r/\alpha)^\gamma - 1]/\gamma \quad (2)$$

where  $\alpha$  and  $\beta$  are scaling constants, and  $\gamma$  is the scaling exponent. The exponent  $\gamma$  was zero for both diamond and the surface polished in antiquity, approximately 0.25 for silica and about 0.75 for alumina.  $\gamma = 0$  indicates a logarithm, demonstrated by the linear appearance of the overlapping curves for diamond and the surface polished in antiquity on the semi-log plot in Figure 5 (b); by contrast, those of the alumina- and silica-polished surfaces increase at faster rates. Thus, the diamond-polished surface clearly quantitatively best matches the surface polished in antiquity. While corundum and quartz abrasives selectively remove the softer diaspore faster than the more durable corundum grains, gouging the surface, the far harder diamond abrasive cuts through both with nearly equal efficacy, leaving a flat surface with little trace of the local microstructure. It is therefore not only the hardness of the corundum in these axes but the way in which it is surrounded by softer diaspore that strongly argues for polishing with a diamond abrasive.

## DISCUSSION

The presence of corundum in these securely dated, excavated early axes from the SXC and BYYY sites of the Neolithic in China *c.* 4000–3500 BC provides the earliest physical evidence world-wide for the use of corundum in a worked object by as much as two millennia. Previously, the



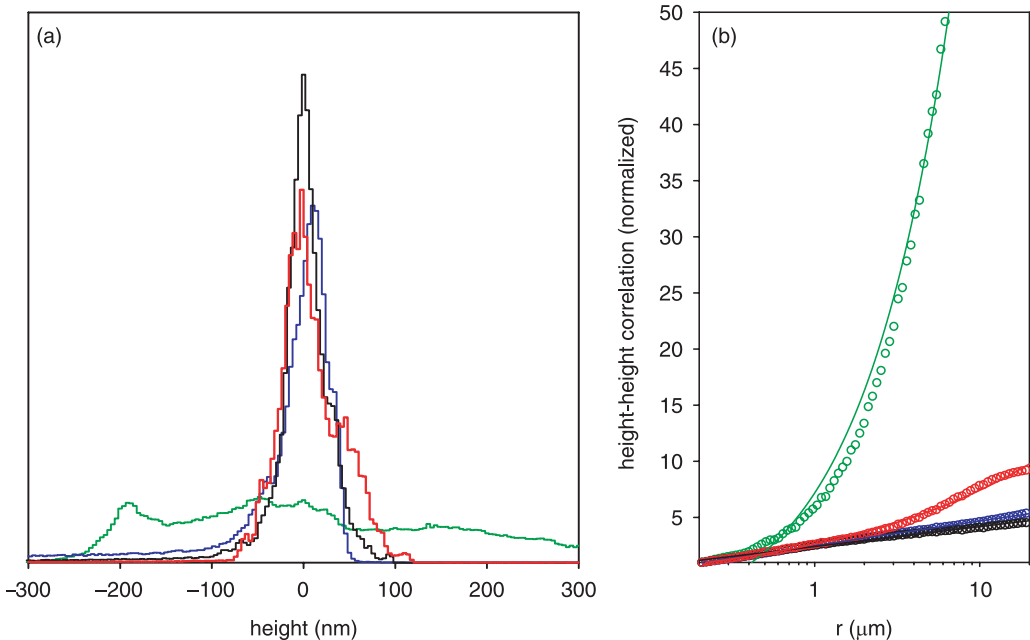


Figure 5 Quantitative functions of the AFM data, demonstrating the closest match between the diamond-polished surface and the surface polished in antiquity. (a) Height histograms of the AFM data displayed in Figure 4. The histogram from the alumina-polished sample (green) is much wider and lower than the other three; the diamond-polished surface (blue) best matches the surface polished in antiquity (black), while the silica-polished (red) distribution is wider and is bimodal, with a shoulder forming a second peak to the right of the primary peak. (b) A semi-logarithmic plot of the normalized, azimuthally averaged height–height correlation functions of the AFM data displayed in Figure 4. The open circles are the correlation function calculated according to equation (1); the solid curves are best-fit curves according to equation (2). Data from the diamond-polished surface (blue) and the surface polished in antiquity (black) closely follow each other and are linear; the silica-polished (red) and alumina-polished (green) surfaces diverge significantly faster, and clearly do not match the surface polished in antiquity.

earliest physical evidence for its use was thought to be the corundum-bearing black powder recovered from bore cores of late Minoan (c. 1700–1500 BC) quartz seals (Heimpel *et al.* 1998). The first reported physical evidence for corundum itself being shaped does not occur until the last quarter of the first millennium BC (Moorey 1994), roughly contemporaneous with the first textual evidence in Theophrastus’ *On stones* (c. 315 BC; Eichholz 1965).

Corundum is not a common rock-forming mineral, and corundum-dominant rocks, such as that from which these axes were fashioned, are very rare. In nature, alumina enrichment to the extent and distribution seen in these rocks is the result of extreme tropical weathering that yields soils and cap rocks with only highly insoluble hydroxides of aluminum and iron remaining. These palaeosols are termed laterite or ‘bauxite’. High-temperature metamorphism of bauxites is required to produce a ‘metabauxite’ rich in corundum. The most well-known variety is emery, a mixture of corundum, iron-rich spinel-family minerals (de Fournestier 1999) and possibly tourmaline, mica and rutile, with diaspore as a primary or subsequent hydration alteration phase (emery is also an abrasive known from antiquity, named from a source near Smyrna [Izmir], Turkey). The YW rock is low in iron content compared to most bauxites, so it

may be the metamorphic equivalent of so-called 'white' bauxite (known from the Balkan region), from which Fe has been removed under reducing conditions during diagenesis (Feenstra 1996, pers. comm. 2004). Moreover, the muscovite content probably represents the introduction of potassium and silica during the corundum-forming metamorphism. The micron-scale grain size indicates relatively low-temperature metamorphism that suppressed grain growth. Texturally, diaspore surrounds corundum grains, suggesting retrograde formation from corundum with relatively recent introduction of water, which is very commonly observed in corundum-rich rocks. We have not found a literature description for a rock like those described here, although laterites and metabauxites are known from south-east China, Vietnam, Laos and Cambodia (Hall and Blundell 1996; Moores and Fairbridge 1997). A potential metabauxite with more than 25% of corundum has been described from Anhui Dabieshan (Sun and Zhou 1994), some 300 km from the Liangzhu site at Hanshan.

Our experimental data from the YW fragment also strongly suggest that diamond abrasive was used by Liangzhu craftsmen to polish corundum axes around 2500 BC. To our knowledge, this is the earliest evidence for man's use of diamond, during the Neolithic. Diamond is thought to have been first known no earlier than 500 BC (Harlow 1998), and used to drill beads from Arikamedu, India, after 250 BC (Gorelick and Gwinett 1988). The earliest securely datable authors to reference what is probably diamond, Manilius and Pliny the Elder, lived in Rome during the first century AD (Healy 1999), although the first historical reference to diamond in China comes more than two centuries later, and its first use as an abrasive is not recorded until the Song Dynasty (AD 960–1278; see Laufer 1915).

The use of diamond by Liangzhu craftsmen is plausible geologically. Two alluvial diamond sources, including the commercial Tancheng placer deposit in Shandong (Deng *et al.* 1996), and a locality further up the Yihe river in Jiangsu (Keller and Wan 1986), have both yielded diamonds in excess of 50 carats and are within 300 km of the SXC and BYYY sites. Alluvial diamonds at either source might have been separated from local gravels using an ancient technique (Laufer 1915): when wet diamond-bearing gravels are run over a greased surface such as a fatty animal hide, the diamonds adhere to the grease while the rest of the rock washes away (Harlow 1998).

## CONCLUSIONS

We find that Neolithic craftsmen of ancient China were certainly using corundum and very possibly diamond about two millennia before anyone else was known to have done so, although further studies may be needed to establish the specific tools and techniques used to create these lustrous surfaces. The availability of corundum and potentially diamond may also help to explain the trademark feature of the Liangzhu lithic industry, amazing quantities of finely polished nephrite jade (Moh's 6.5) artefacts with intricate carved decoration. Quartz, with slightly greater hardness and wide abundance, has been generally assumed to have been the major abrasive used in prehistoric China (Rawson and Ayers 1975). Our findings support the possibility that the Liangzhu lapidary's workhorse abrasive could have been corundum. More abundant than diamond and far harder than nephrite and quartz, corundum abrasives could have significantly increased cutting rates and concomitantly decreased production time. And diamond may have provided the finishing touches that made these jades the most prized objects of their time (Hayashi 1996). For the final lustrous polish, the Liangzhu worker might have turned to diamond powder, and for incised embellishments, to individual diamond points.

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