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Yoshiyuki Iizuka, Peter Bellwood, Ipoi Datan and Hung Hsiao-chun

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Non-invasive<sup>1</sup> mineralogical studies of deep green jade earring (*lingling-o*) from Niah Cave West Mouth in Sarawak have been conducted using a low-vacuum scanning electron microscope with an energy dispersive x-ray spectrometer. The Niah earring is identified as nephrite (tremolite-actinolite) with zinc bearing chromian-spinel (chromite) inclusions. The mineralogical characteristics of this nephrite are very close to those of Fengtian nephrite from eastern Taiwan, this source being located approximately 2500 km to the north of the Niah Caves.

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## FORMAL DESCRIPTION OF THE STUDIED SAMPLE<sup>2</sup>

The Niah *lingling-o*<sup>3</sup> (Fig. 1) was excavated on 29<sup>th</sup> March 1977 during archaeological investigations by Zuraina Majid in Niah Cave West Mouth, Sarawak, East Malaysia. It was found in square M/D3 at 6 inches depth below the surface (Chin, 1980: Fig. 2.14; Zuraina, 1982: 46 and 2003: 198), and was recorded by Zuraina as having been found with sherds stratified seven inches above a burial of unspecified date. This specimen is a penannular earring with three pointed circumferential projections. The matrix has a deep green colour, and the inclusions are black to dark brown. The maximum diameter of this piece is 31.0 mm, thickness 16.0 mm; hole diameter 9.0 mm (outer) and 7.0 mm (inner); slit width 1.8 mm; weight 9.0 gm. The three necked and pointed circular-sectioned projections extend for 4.5 to 5.0 mm beyond the outside of the ring. The date of this specimen is not evident from its burial context, but similar

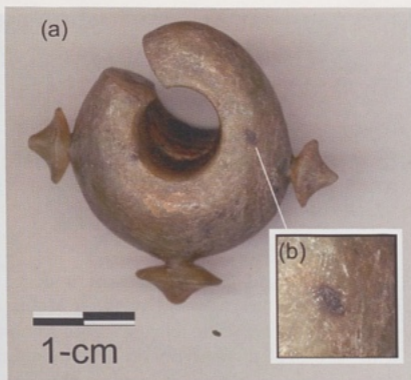


Fig. 1: (a) The *lingling-o* from Niah Cave West Mouth (NC-J6), Sarawak Museum; (b) an enlarged photograph of Cr-spinel grain.

complete specimens from the Philippines are known to be of Iron Age date, presumably manufactured between 2000 and 1000 years ago (Fox, 1970: 131).

## ANALYTICAL METHODS

The analytical procedure used was the same as that described for the analysis of Philippine jade artifacts by Iizuka *et al.* (2005). Before observation, the sample was well cleaned repeatedly for a few hours in an ultra-sonic bath with distilled water to remove dust and soil from its surface. It was then rinsed in ethanol, and dried in air overnight at 75°C.

A scanning electron microscope (JEOL JSM-6360LV) was used with 15 kV acceleration voltage and 0.18 nA electron beam current, under low-vacuum conditions (25 Pascal). Surface observation and chemical analysis were conducted without the use of gold or carbon

coatings. The analysed points were selected on the relatively flat and well-polished surface of the artifact under the back-scattered electron image. The chemical compositions (greater than 0.5% by weight) of the matrix and inclusion minerals were analysed by EDS (Oxford Instruments INCA-300) at the 1  $\mu\text{m}$  electron beam spot and results were corrected against the X-ray intensities of standard minerals. The number of cations was calculated as atoms per formula unit on the basis of 23 oxygen atoms, and minerals were classified according to the nomenclature of amphiboles (Leake *et al.*, 1997). The corrected data in the cation ratios are based on the ideal chemical formula of calcium amphibole  $[\text{Ca}_2(\text{Mg,Fe})_5(\text{Si}_8\text{O}_{22})(\text{OH})_2]$ ; where a ratio of  $\text{Mg}/(\text{Mg} + \text{Fe})$  less than 0.9 indicates actinolite; greater than 0.9 indicates tremolite.

## RESULTS

The surface of the *lingling-o* was well-polished by ancient craftsmen and was therefore no problem to analyse. Fibrous textures were observed on the surfaces of the green coloured matrix at all points studied (Fig. 2). A representative X-ray spectrum by EDS from the matrix is shown in Fig. 3. The quantitative chemical data are shown in Table 1 and plotted in Fig. 4 as a discrimination diagram of calcium (Ca) amphiboles [ratio of Si to  $\text{Mg}/(\text{Mg} + \text{Fe})$ ]. The matrix portion can be identified as both tremolite and actinolite in the Ca-amphiboles. Based on the chemistry and surface texture, the Niah *lingling-o* is of nephrite.

Chromium (Cr)-spinel, black in colour, were also observed at the surface of the specimen as inclusion minerals (Figs. 1b and 5). The sizes of these inclusions vary from a few  $\mu\text{m}$  to 2 mm. A zoned inclusion, spinel-rich ( $\text{AlMg}_2\text{O}_4$ ) in the inner part and chromite rich ( $\text{CrFe}_2\text{O}_4$ ) in the outer part, can be observed in the back-scattered electron image (Fig. 5). A representative EDS spectrum from a Cr-spinel (chromite) inclusion is shown in Fig. 6. It shows strong intensities of iron (Fe) and chromium, and minor peaks of manganese (Mn) and zinc (Zn). The highest value recorded for ZnO was 4% by weight. Based on these results, the inclusion can be termed Zn-chromite.