New Developments in Pearl Analysis : X-ray Micro Tomography and Radiocarbon Age Dating

Michael S. Krzemnicki[1], Sebastian Friess[2], P. Chalus[3], Irka Hajdas[4], Henry A. Hänni[5]

[1] *SSEF Swiss Gemmological Institute, Switzerland, email: gemlab@ssef.ch*
[2] *Gloor Instruments AG, Uster, Switzerland*
[3] *F. Hoffmann-La Roche AG, Basel, Switzerland*
[4] *Laboratory of Ion Beam Physics, Radiocarbon Dating, ETH Zurich, Switzerland*
[5] *Research Associate of SSEF Swiss Gemmological Institute*

近期瑞士寶石研究基金會實驗室(SSEF)接到大量珍珠進行鑑證，因無核養珠的培植及發展令到天然珍珠及養植珍珠之鑑證更加困難。SSEF近期發展兩個鑑證項目，對珍珠進行更深入的研究。它們是：

X射線微斷層掃描

放射性碳^{14}C年代分析

Recently, the SSEF Swiss Gemmological Institute has tested a large number of natural pearls as they attract more and more interest in a specialised segment of the jewellery market. However, separating natural pearls from cultured pearls is not easy and has become even more difficult with the number of beadless saltwater cultured pearls being sold nowadays under the term "new Keshi". Commonly, this separation of pearls is based mainly on analyses of radiographs (and in rare cases X-ray diffractograms), X-ray luminescence, and meticulous microscopic observations.

Two projects have been launched recently at the SSEF in order to gain more insight into the nature of pearls:

- X-ray micro tomography

- Radiocarbon (^{14}C) age dating

X-ray micro tomography:

Computerised X-ray micro tomography was developed in the 80s. The analysis consists of two steps. First, the sample is exposed to a focused X-ray beam and rotated. Step-by-step, the X-ray absorption is registered (similar to radiography) on a planar X-ray detector. And, second, these projected images are used to calculate a three dimensional reconstruction of the sample being investigated. Both, X-ray scanning and reconstruction are quite time- and megabyte consuming. The computerised tomographical reconstruction can then be scrolled through on three orthogonally oriented virtual slices. Furthermore, distinct X-ray absorption contrasts within the sample can be calculated into a 3-dimensional model, which can be rotated in all directions (see figure 1). Summarising, this method provides a very visual approach to analysing the internal structures of solids. It has largely been used for quality analysis of industrial products (e.g. medicaments, foams) and in-vivo medical research (e.g. bones). It is only recently that X-ray micro tomography has been applied to pearl [1, 2, 3] and gemstone analysis [4].

We analysed several natural and cultured pearls (beadless and beaded), with a special focus on beadless saltwater cultured pearls (SWCP) of the so-called "new Keshi"-type. We used a Skyscan 1172 instrument, equipped with a Hamamatsu X-ray tube and an 11 Megapixel camera. With this instrument and our setup, a single loose (or mounted) pearl was analysed in about 20 - 100 minutes, depending on the chosen analytical parameters.

Apart from obvious cases of beaded cultured pearls, the method also proved very successful for the identification of beadless cultured pearls. These pearls are characterised by an internal cavity, often in the shape of a small "umbrella" or "moustache", representing either the shape of the crumbling implanted epithelium tissue or the shape of the collapsing pearl sack. Even in cases without any evident internal cavity structure on radiographs, X-ray micro tomography enabled us to scroll through the tiny internal cavity virtually after the analysis.

Based on these positive results, the SSEF has now implemented X-ray micro tomography as an additional service in our pearl testing routine if the separation of natural pearls and cultured pearls is not feasible by classical radiography.

Radiocarbon ^{14}C dating of pearls and shells:

Nowadays the radioactive ^{14}C carbon isotope is widely used for radiometric dating of carbon rich (especially organic) materials in both, archaeology and Earth Sciences. The method is based on the principle that a living organism (animal or plant) is constantly equalising its carbon isotope ratio (ca. 99% ^{12}C, ca. 1% ^{13}C, and ca. 1,2 x 10^{-12} radioactive ^{14}C) by metabolism with the surrounding (atmosphere). After its death, this process is stopped and due to the radioactive decay of ^{14}C the carbon isotope ratio will change and thus the age of the organism can be calculated.

The first ^{14}C ages produced 60 years ago on archaeological and geological samples of "known age" illustrated the potential of the new method for these research fields [5]. Usually, the method is used for age determination in the range of 300 – 50,000 years. However, the so-called "bomb peak", i.e. the excess of ^{14}C produced artificially during the 1950s/60s nuclear tests and the subsequently monitored flattening out of this ^{14}C peak, enable us to date samples even to very recent ages [6].

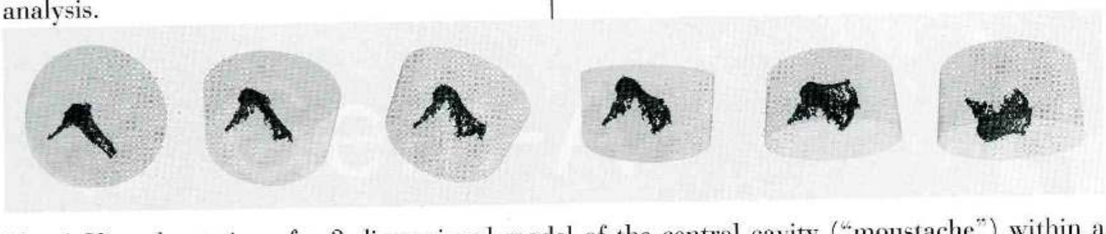

Fig. 1 Virtual rotation of a 3-dimensional model of the central cavity ("moustache") within a beadless cultured pearl (sample MXT-1) calculated from X-ray micro tomography data.

For this study, we are interested in the feasibility of radiocarbon age dating of pearls. We are especially interested in discovering whether recently grown pearls can be separated from historical ones. For our preliminary study, we have selected a number of pearls of very recent growth (2001) to approximately 1950 (SSEF collection) and shells of historical ages (the beginning of the 20th century to mid 17th century), documented by the Natural History Museum of Basel (Switzerland). By using the accelerated mass spectrometer (AMS) at the Ion Beam Laboratory of the ETH Zurich, it is possible to carry out radiometric age dating on just a few milligrams of sample material. Thus, our pearl samples were partially drilled and slightly polished and the resulting carbonate dust was collected for analysis.

The authors presented the first results of their ongoing ^{14}C study during the IGC conference in Arusha in October of this year.

References:

[1] Wehrmeister U., Goetz H., Jacob D.E., Soldati A., Xu W., Duschner H., and Hofmeister W. (2008) Visualization of the Internal Structure of Freshwater Cultured Pearls by Computerized X-Ray Micro Tomography. *Journal of Gemmology*, 32, 1/2, 15-21.

[2] Wehrmeister U., Soldati A., Goetz H., Jacob D.E., Xu W., Duschner H., and Hofmeister W. (2009) Röntgen-Mikro-Computertomographie: Zerstörungsfreier Einblick in das Innere von Zuchtperlen. *Zeitschr. Dt. Gemmol. Ges.* 58/1-2, 53-66.

[3] Hänni H.A. (2008) ^{14}C Age Determination of "Neptunian" Beads from Asia. *Gems & Gemology*, Fall 2008, p. 201-294.

[4] Hänni H.A. (2009) How Much Glass is in the Ruby? *SSEF Facette*, No. 16, p. 8-9.

[5] Arnold J.R., Libby W.F. (1949) Age Determinations by Radiocarbon Content: Checks with Samples of Known Age. *Science* 110 (2869), 678–80.

[6] Hajdas, I. (2009) Applications of Radiocarbon Dating Method. *Radiocarbon*, 51, 1, 2009, 79-90.

Acknowledgements:

A. Puschnig from the Natural History Museum for the donation of historical shells.

Pearls were donated or loaned by Mr. R. Totah and T. Chantitch.

Dr. L. Adamy (F. Hoffmann-La Roche AG, Basel, Switzerland) and Dr. G. Bonani (Laboratory of Ion Beam Physics, Radiocarbon Dating, ETH Zurich, Switzerland) for their support in using their facilities.