Case Studies in Nondestructive Testing and Evaluation 6 (2016) 1–7

Contents lists available at ScienceDirect

Case Studies in Nondestructive Testing and Evaluation

www.elsevier.com/locate/csndt

Characterization of pearls by X-ray phase contrast imaging with a grating interferometer

Vincent Revol [a,*], Carina Hanser [b], Michael Krzemnicki [b]

[a] CSEM SA, Untere Gründlistrasse 1, 6055 Alpnach Dorf, Switzerland
[b] SSEF Schweizerisches Gemmologisches Institut, Aeschengraben 26, 4051 Basel, Switzerland

ARTICLE INFO

Article history:
Available online 8 June 2016

ABSTRACT

In this study, X-ray phase contrast imaging with a grating interferometer is applied on pearls for the first time in order to distinguish natural pearls from cultured pearls. Traditionally, this separation is mainly based on X-ray radiography. In order to visualize the internal structure of pearls we used a custom-made grating interferometer setup and performed measurements on three different pearl products, a natural pearl, a beaded cultured pearl and a beadless cultured pearl. To enhance the visibility of the internal pearl structures, we applied a high-pass filter in order to better conclude on the applicability of this technique to the separation of natural and cultured pearls. The study shows that it is possible to visualize internal pearl structures using distinctly shorter exposure times compared to traditional X-ray radiography and that X-ray phase contrast imaging is a promising complementary method for pearl analysis.

1. Introduction

One of the major issues in the international pearl trade is the reliable separation of natural pearls from cultured ones. Natural pearls accidentally form in a wild mollusc without any human intervention, whereas cultured pearls result from a grafting operation in certain mollusc species (e.g. *Pinctada maxima, Pinctada margaritifera, Unio*) [1–4]. Generally, there is an important price gap between these two products in the trade. In 2011, the Peregrina pearl, a historical natural pearl of exceptional size and quality, was sold for US$ 11 mio at auction, whereas cultured pearls of low quality may just cost a few cents [5].

The trade relies on specialized gemmological laboratories to identify pearls and to distinguish natural from cultured pearls. This separation requires a detailed analysis of the pearl's internal structure, such as the presence, concentration and orientation of organic matter within the calcium carbonate matrix. Based on this analysis, it is possible to trace back the natural or cultured formation of a pearl. Few non-destructive techniques are capable of achieving a sufficient level of details to perform an efficient separation. Most commonly, pearl testing is performed using X-ray film techniques [2,6]. The method is, however, time-consuming and requires the use of chemicals. X-ray digital radiography is still confronted with technical challenges such as the sensitivity, dynamic range and resolution of detectors – in particular because of the spherical shape of the pearl. The variable path lengths within the pearl lead to strong variations of the X-ray intensity on the detector.

* Corresponding author. Tel.: +41 41 672 7533, fax: +41 41 672 7500.
E-mail address: vincent.revol@csem.ch (V. Revol).

http://dx.doi.org/10.1016/j.csndt.2016.06.001

Fig. 1. (From left to right): saltwater natural pearl (NP-2f), beaded saltwater cultured pearl (CP-2f) and beadless freshwater cultured pearl (CP-1b).

The use of an immersion liquid with matching X-ray attenuation helps to reduce these artefacts but cannot avoid them. Moreover, the usual matching liquids are difficult to handle as a result of their toxicity.

In recent years, digital X-ray micro-tomography (µCT) has been introduced to visualize the internal structure of a pearl in three dimensions (3D). Indeed, pearls do not only consist of $CaCO_3$ but also of organic layers which can be distinguished because of their differing X-ray densities. Similarly, beads and cavities can be detected [7,8]. Although µCT allows a better evaluation of the orientation of such features compared to X-ray radiography, it requires long exposure times (from 30 to 120 minutes) and generates large data sets, which renders the data analysis complex and time-consuming. There is thus a need for novel non-destructive techniques, capable of revealing the internal structure with short measurement times.

Within this collaboration, we have investigated the potential of using grating-based X-ray phase contrast imaging for the characterization of pearls. The technology has initially been developed using synchrotron light [9,10] and was later on extended to be usable with standard X-ray tube sources [11]. Recently, technical improvements on the design of X-ray interferometers allowed to extend the use of this technique toward industrial applications by enlarging the size of the field of view and the usable X-ray energies [12–14].

We report here on the analysis of X pearl samples of different types and discuss the applicability of the method for detection of natural versus cultured pearls.

2. Materials and methods

2.1. Test specimen

For this study, we selected three pearl samples representing the three main pearl types according to their formation: a natural pearl, a beaded cultured pearl and beadless cultured pearl (Fig. 1).

- NP-2f is a small, slightly drop-shaped saltwater natural pearl of cream colour from Pinctada radiata and originates from the Arabian Gulf in Bahrain.
- CP-2f is a large, round, beaded saltwater cultured pearl from Pinctada margaritifera. It is brownish to brownish-grey in colour, shows irregular surface structures and originates from Tahiti.
- CP-1b is a white, button-shaped beadless freshwater cultured pearl from Hyriopsis cumingii and was produced in China.

2.2. Grating interferometer

The grating interferometer used for the experiments consists of the combination of a commercial X-ray source tube (Varian HPX160-20, focal spot 0.4×0.4 mm^2), a commercial X-ray detector (Dexela DEX2315, pixel size 75 µm, resolution 3072×1944) and three custom-made X-ray gratings. A schematic view of the setup is displayed in Fig. 2.

The three gratings were produced in the clean rooms of CSEM in Neuchâtel, Switzerland using silicon wafers with a diameter of 150 mm. The phase grating G1 was made out of silicon by wet etching while G0 and G2 are absorption gratings obtained by gold deposition on a structured silicon substrate. The periods p_0, p_1 and p_2 of the three gratings G0, G1 and G2 were equal to 20 µm, 3.333 µm and 4 µm respectively, while the depths were equal to 100 µm, 25.77 µm and 100 µm respectively. The depth of the grating G1 made out of silicon corresponds to a phase shift of $\pi/2$ for the design X-ray energy of $E_d = 40$ keV. The distance between gratings G0 and G1 was 1075 mm and between G1 and G2, 215 mm. Because of the magnification of 1.3 used here, the intrinsic resolution in the sample was equal to 57.7 µm.