X-ray phase contrast and X-ray scattering images of pearls

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Introduction

The separation of natural pearls from cultured pearls is mainly based on the analysis and observation of their internal structures. Due to their value and their importance as a cultural and even archaeological heritage, it is absolutely mandatory that such testing is non-destructive. Traditionally, pearl testing is mainly based on X-ray radiography (Anderson 1931, Strack 2006, Sturman 2009), visualizing slight variations in X-ray absorption within a pearl. These variations are linked to the presence, concentration, and orientation of organic matter or voids within the calcium carbonate pearl matrix. In recent years, X-ray microtomography (Xray-µCT) has strongly contributed to a better understanding of the spatial distribution of such internal features (Wehrmeister et al. 2008, Krzemnicki et al. 2010). In the present study, we have investigated the potential of X-ray phase contrast and X-ray scattering as new and promising complementary methods for pearl analysis.

Instrumentation and samples

Conventional X-ray imaging (radiography, CT scans) relies on the absorption of X-rays (attenuation) when transmitted through a sample material but does not take into account the phase shift of the X-ray beam caused by the sample. One of the main drawbacks in conventional X-ray imaging is the limited possibility to increase the absorption contrast, especially for materials of similar absorption properties (e.g. biological samples) (Zhu et al. 2010).

Although widely used in gemmological laboratories, X-ray radiography has a number of limiting factors due to instrument parameters (e.g. focal size of the X-ray tube), analytical geometry (X-ray exposure is cone-shaped, resulting in lateral geometrical distortions of any 3D-object on the detector) and dynamic range and resolution of detectors (or X-ray films). But the most limiting factors are intrinsic properties of the pearl, such as its spherical geometry and thus variable path length (and attenuation) of the transmitted X-rays, and the tiny dimension and geometric orientation of internal structures (e.g. organic matter in the centre of the pearl).

By using a combination of gratings it is possible to build an X-ray interferometer and obtain X-ray phase contrast images, for which the beam phase shift is transformed into intensity variations, which are then recorded by a detector. Phase contrast X-ray imaging substantially improves contrast information compared to conventional attenuation-based methods. Especially for biological samples (e.g. tissues) made up of low-Z elements, the phase contrast effect is much more pronounced than the X-ray attenuation (Zhu et al. 2010).

Based on ground-breaking research on synchrotron X-ray interferometry by research groups at the Paul Scherrer Institute (PSI) in Switzerland and the University of Tokyo (David et al. 2002**,** Momose et al. 2003), Pfeiffer et al. (2006) were able to transfer the grating-based method to conventional laboratory X-ray tubes. The same research group developed an additional imaging method, using the X-ray signal scattered at microstructures of the sample (Pfeiffer et al. 2008).

By using an experiment setup at the Centre Suisse d'Electronique et Microtechnique (CSEM) (Revol et al., 2011) we were able to simultaneously register conventional X-ray absorption, X-ray phase contrast, and X-ray scattering images of numerous natural and cultured pearl samples. Not only was it possible to analyse with our setup single loose pearls, but also pearls strung on a strand and to display such a strand in one image. For our study, we used the following samples: natural pearls from *Pinctada radiata* and *Pinctada maxima*, beaded cultured pearls from *Pinctada margaritifera* and *Pinctada fucata,* Tokki cultured pearls (Krzemnicki et al. 2011) from *Pinctada margaritifera*, beadless cultured pearls from *Hyriopsis cumingii*, and Keshi cultured pearls (beadless) from *Pinctada maxima* and *margaritifera*.

Furthermore, a natural pearl from *Pinctada maxima* from Australia was selected to register absorption, phase contrast and scattering CT scans (microtomography).

Results and discussion

Our preliminary results show that both, X-ray phase contrast and X-ray scattering are adding valuable information to conventional X-ray absorption when analysing the internal structures of pearls.

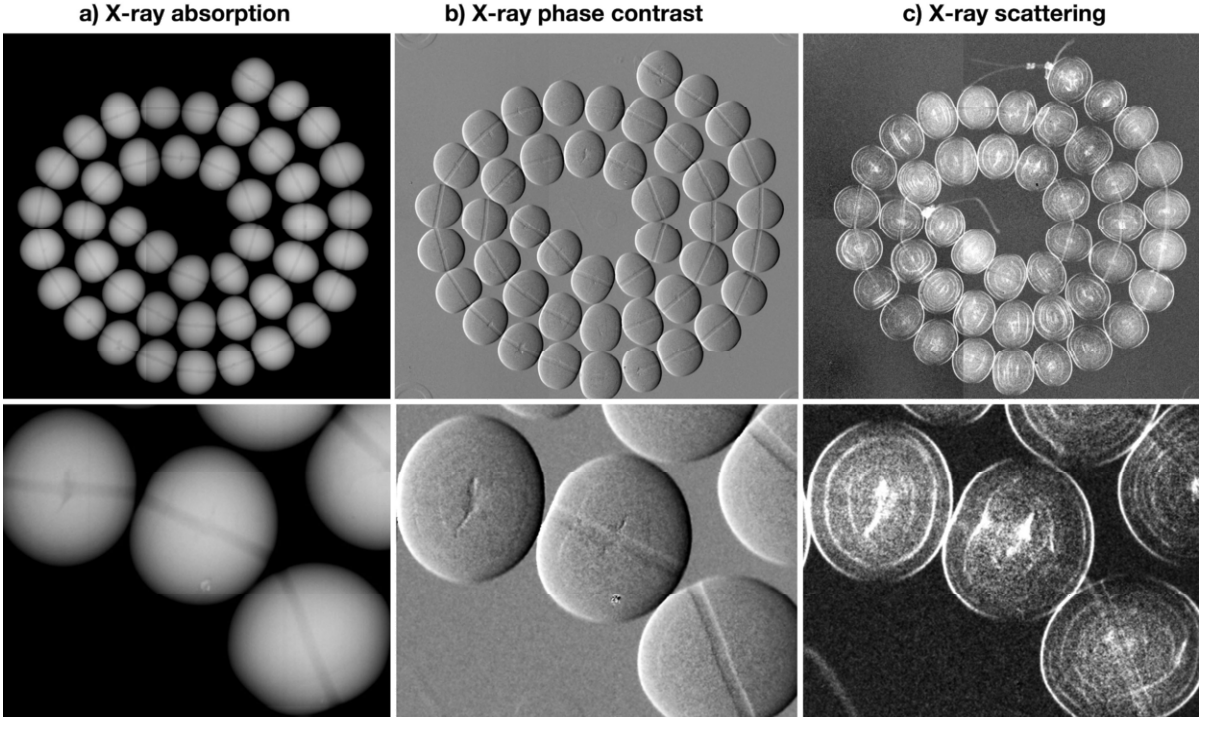

Figure 1. Comparison of a) X-ray absorption (attenuation), b) X-ray phase contrast, and c) X-ray scattering images of a strand of 44 beadless freshwater cultured pearls from China (Hyriopsis cumingii) as the whole necklace and magnified.

With our setup with a large focal spot size (1x1 mm^2), the absorption radiographies showed a rather low contrast, compared to conventional digital radiography using a microfocus X-ray tube. So when testing a strand of beadless Chinese freshwater cultured pearls from *Hyriopsis cumingii*, their characteristic comma-shaped central structure rich in organic matter (Strack 2006) was hardly visible (Figure 1a). Even though the absorption gradient between the organic matter (conchiolin) in this central structure and the surrounding calcium carbonate is very pronounced. Apart from the instrumental limitations with our setup, this is also due to the nearly two-dimensional geometry of this structure and to its random orientation within the centre of these cultured pearls in respect to the X-ray beam.

The phase contrast image of the same strand reveals a virtual morphological pattern of these pearls superposed by a relief-shading effect at the outline of the pearls and along the drill hole (Figure 1b). The very fine onion-like growth layers of these beadless cultured pearls are hardly discernible, in contrast to the partly marked visibility of their characteristic central feature.

However, the most detailed information is gained from the X-ray scattering image (Figure 1c), which in fact reveals not only the multi-layered onion-like growth structure of these cultured pearls, but also in great detail the complex geometry of the characteristic central structure of these beadless cultured pearls. Although a number of image processing software is available, superposing the different X-ray images and adding colour conversions, already a simple greyscale inversion is very helpful (Figure 2). It readily enables a laboratory gemmologist to compare an X-ray scattering image with a conventional absorption radiograph, in which less dense and organic rich zones are darker compared to nacre.

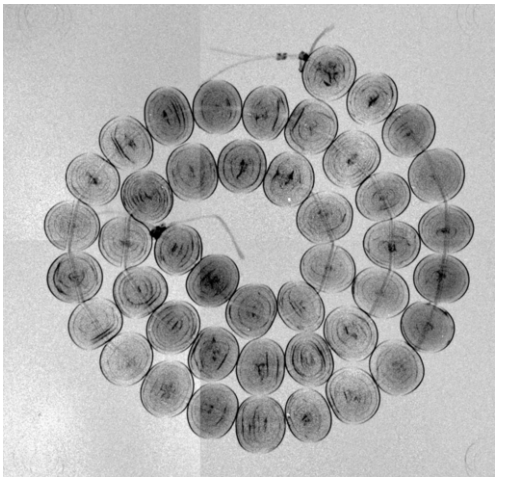

Figure 2. Simple inversion of the X-ray scattering image results makes comparison with conventional X-ray absorption images more easy.

In analogy to the above stated observations, the X-ray images of the other tested pearls (natural and cultured) revealed very similar results. Again, the X-ray scattering images were by far the most promising in terms of visualisation of internal structures of the studied pearls.

Finally, a natural pearl from *Pinctada maxima* (7.58 ct) was chosen for CT scans, using X-ray absorption, phase contrast and scattering. Compared to a conventional CT scan using a microfocus X-ray tube, this first attempt is still rather unsatisfactory due to its low resolution and rather high noise signal. Nevertheless the obtained X-ray scattering CT data was rendered into a three-dimensional virtual model of this natural pearl, enabling us to see its internal structures during rotation.

Conclusions

Preliminary results of our study on the application of new X-ray imaging methods using phase contrast and X-ray scattering show that both methods, but particularly X-ray scattering, add valuable information to conventional absorption radiography.

Despite the current drawbacks at this level of research, we presume that these new X-ray imaging techniques - including CT scans based on X-ray scattering – will become important for pearl analysis in the near future when commercial instruments become available.

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