Illuminating the past with high-resolution light microscopy

Markus Fabich

Discovering and understanding novel strategies for historic conservation, Dr Olivier Schalm's laboratory at the University of Antwerp is benefiting from the latest in high-resolution light microscopy - as Product and Application Specialist for Materials Science Microscopy at Olympus, Markus Fabich, explains.

Revealing details of the microstructure that otherwise remain hidden from the naked eye provides unique insights of historical materials – from discovering an artefact's history to deciding the best course of action for its future preservation.

Light microscopy is therefore a popular analytical tool within historic conservation: only by truly understanding the mechanisms of degradation can one develop and optimise techniques to control and slow the process, or restore an object to a past state. At the Department of Conservation Studies, University of Antwerp (Belgium), these aims form the major focus of Dr Olivier Schalm's research efforts.

Previously, if an investigation called for greater resolution, Dr Schalm would often turn to scanning electron microscopy (SEM), but this is now changing. The latest light microscopes are capable of reaching far higher resolutions, with magnifications of up to 4,000x now possible. In many cases, magnifying above this level is unnecessary, and with light microscopy not only retaining true-colour information but also utilising the latest digital imaging technologies, this approach can often present an attractive alternative to SEM. The recent introduction of one such system – the Olympus DSX500 digital light microscope – into Dr Schalm's department has facilitated many studies, including analysing damage on antique photographic plates, advancing the understanding of metallic degradation and probing the corrosion of glass.

Preserving the roots of photography

Introduced in 1839, the daguerreotype was the first mainstream photographic process, with silver and mercury nanoparticles forming the image upon a silver surface. Being susceptible to scratches and tarnishing, understanding silver corrosion is vital for optimising cleaning treatments and preservation, but the damage must first be characterised.

One important aspect of analysing the damage of antiques is knowing where a feature sits within the context of the whole piece, as microstructures can differ significantly across the artefact. Every point on such an object is unique and this link is vital, and should be retained when investigating the sample at higher magnifications. Navigation is often achieved using cues from specific features, for example locating a scratch using the feature of the eye as a point of reference. When comparing brightfield visualisation at 555x magnification with conventional secondary electron SEM imaging, the image of the eye is only visible with light microscopy. In order to observe a similar feature in a SEM, backscattered electron imaging (where contrast is based on a material's atomic number) needs to be used, which is a more expensive technique.

Rewinding time: reversing corrosion

With modern conservation techniques, it is now possible not only to stop corrosion, but in some cases, actually reverse the oxidation state (i.e. metallic tarnish). This is achieved through atmospheric plasma afterglow treatments, and the efforts of Dr Schalm's research group in improving this technique are part of the European PANNA project ("Plasma and nano for new-age soft conservation").

Whilst pure silver is easily reduced, copper is impervious to current plasma treatments, and unfortunately, historical silver often contains a small amount of copper (e.g. sterling silver), yet the basis for this remains unknown. A greater understanding is not only vital for improving plasma treatments for

Light microscopy in historic conservation

Understanding the past

- Placing an artefact in its socio-cultural context – how/when/where was it created?
- Identifying, analysing and interpreting the component materials
- Understanding the process of degradation

Deciding the future

- Preventing degradation by manipulating the storage environment
- Choosing the best intervention for conservation or restoration
- Ongoing evaluation of protective measures

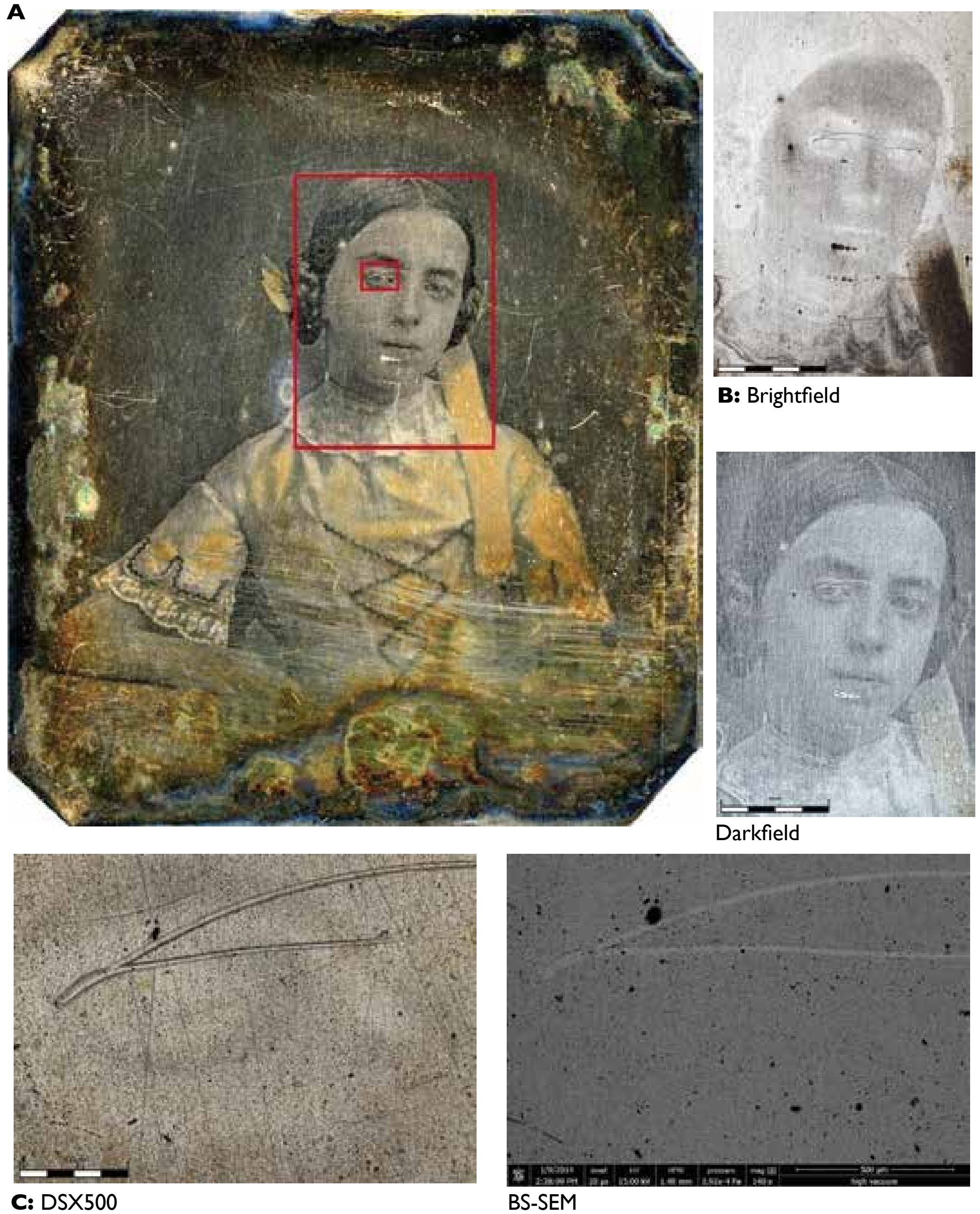


Figure 1: Understanding daguerreotype degradation. *The silver plate (A) was analysed with darkfield (positive) and brightfield (negative) (B, Field of view 4mm). When locating a scratch, compared to backscattered scanning electron microscopy (BS-SEM, Field of view 500µm) , light microscopy at 555x magnification delivered the contrast necessary to visualise the eye - a navigation feature. (C, Field of view 400µm). Images courtesy of Dr Olivier Schalm and Eva Grieten, University of Antwerp.*

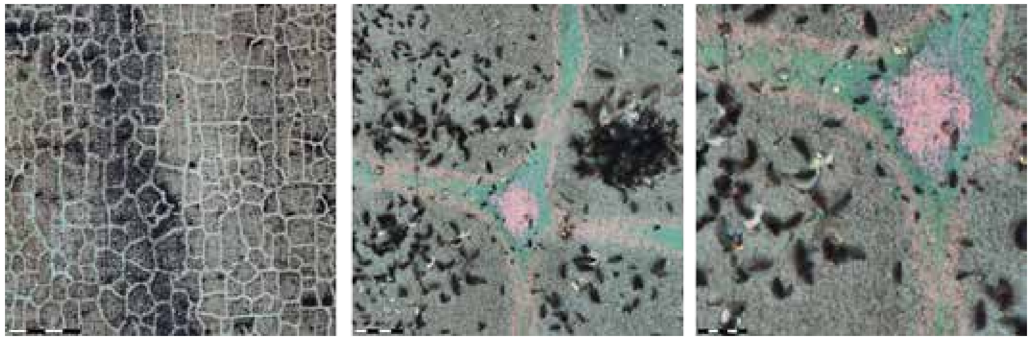


Figure 2: Visualising copper corrosion. *Copper sulphidised in an aerated Na_2S solution was visualised using light microscopy after 30 minutes at magnifications of: 150x (A), 2,000x (B) and 4,000x (C). Retaining true colour information revealed green corrosion at the metallic grain border. Images acquired with the Olympus DSX500; courtesy of Dr Olivier Schalm and Patrick Storme, University of Antwerp.*

different materials, but also in avoiding unexpected damage being caused in the long-term.

Analysing copper corrosion in more depth, a copper plate was immersed in an aerated sodium sulphide solution and the formation of copper sulphide compounds inspected with high-resolution light microscopy. Although SEM generates highly detailed images, these lack colour information, and high-resolution light microscopy has revealed new insights – with Figure 2 showing particles of a corrosion product forming preferentially at the metallic grain border. As a colour rather than morphological feature, these zones of green corrosion were previously missed using SEM, and this discovery was only possible with true colour information. With high-resolution light microscopy allowing researchers to investigate corrosion in unprecedented detail, it has become evident that the chemical reactions involved are more complex than first envisaged a couple of years ago.

A new look at glass corrosion

Although glass begins life as a homogenous and transparent substance, over time it becomes gradually more opaque and irregularities begin to appear. Since Sir David Brewster's efforts in the 19th century, this process was thought to be fully understood. However, looking at glass artefacts in more detail has shown us that this process is perhaps more complex than previously thought, and Figure 3 illustrates an historic glass sample exhibiting an unusual characteristic. Running right through the structure are lamellae formations, and it remains unknown as to how they form, including reasons for the difference between the black and white rings, and the basis for the different results generated from brightfield and darkfield illumination. Detailed analysis over a large area of the sample is crucial for these ongoing studies, and image stitching enables the glass to be viewed at a magnification of 555x over a field of view of 1.4 mm^2. Moreover, like most antiques, the surface of old window glass samples is never perfectly flat, and in the past, generating such sharp images at high magnification was not possible with light microscopy. By creating a z-stack with the Extended Focal Image software function, it is now possible to acquire sharp images of antique samples, enabling detailed analysis and facilitating our understanding of how these irregularities form.

Employing different illumination techniques is also useful for visualising different forms of glass corrosion, such as the formation of manganese-rich inclusions. The glass artefact shown in Figure 4 has developed these inclusions over the last 200 years or so, appearing in dendritic formations. While brightfield observation displays the inclusions on the surface due to a higher refractive index, visualisation using darkfield illumination reveals them beneath the surface. This unexpected finding indicates an intricate three-dimensional structure, without the

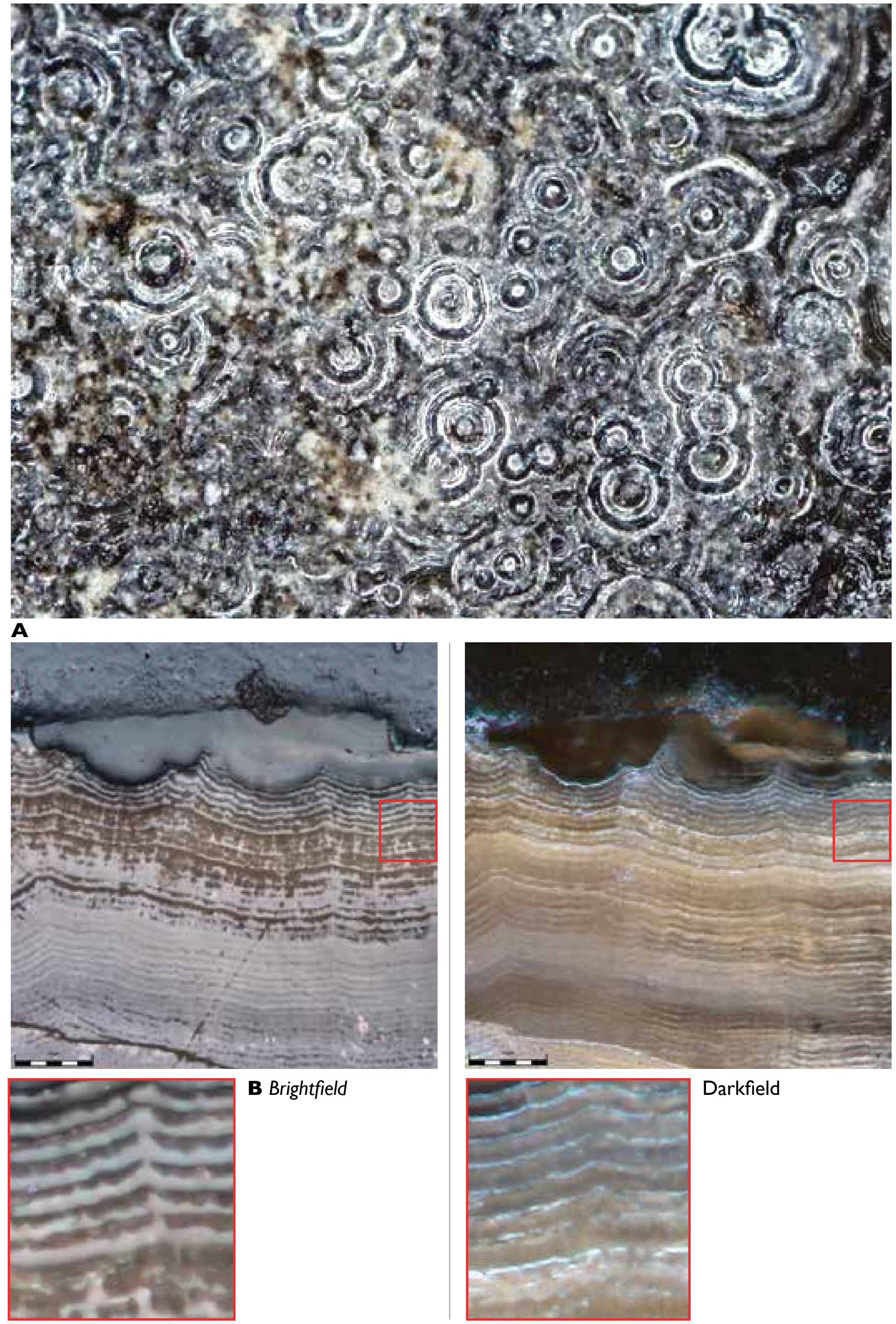
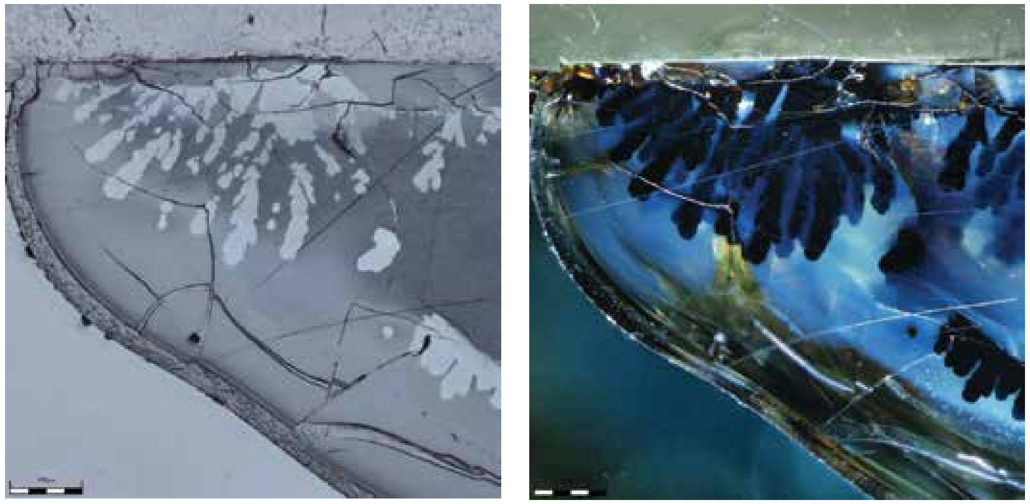


Figure 3: Inspecting the degradation of glass. *This antique glass artefact has become opaque and heterogeneous. A) Surface of a window glass fragment - visualising lamellae in unprecedented detail was achieved with darkfield illumination at 555x magnification over a field of view of 1.4 mm^2, employing image stitching and Extended Focal Imaging capabilities to form a large, fully-focused image. B) Cross section of the sample fragment shows the lamellae running internally, differentially illuminated with brightfield and darkfield at 1,000x magnification, providing further insights into these structures. Images acquired using the Olympus DSX500; courtesy of Dr Olivier Schalm, University of Antwerp*



Brightfield Darkfield

Figure 4: Investigating the formation of mineral inclusions in glass. *Manganese inclusions formed during the last 200-years are visualised with brightfield and darkfield illumination at 100x magnification. Employing the Extended Focal Imaging function creates a fully focused image, and combined with darkfield visualisation shows how the dendritic inclusions are not only superficial, instead running beneath the surface. Images acquired using the Olympus DSX500, 200µm; courtesy of Dr Olivier Schalm, University of Antwerp.*

need for complex tomography experiments. Since this form of manganese is non-mobile, it is suspected that a water-soluble form first travels towards certain regions where a redox reaction takes place, and research aims to elucidate this process further.

In summary

Affording several major advantages, high-resolution light microscopy has enabled Dr Schalm's research group to easily extract intriguing new information from a range of historical artefacts - even presenting a fast and efficient alternative to scanning electron microscopy.

There is still a way to go before our understanding of material corrosion is complete, but moving into the future it is clear that the latest high-resolution light microscopy will play an important role, rapidly advancing our knowledge of the past.

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