The quality of a TEM sample is an important factor when studying the changes in corrosion phenomena in historical photographs. This material has a complex structure made out of a soft matrix with embedded image and corrosion particles. To determine the optimal sample preparation method 2 techniques are evaluated; the classical ultra-microtome and the high tech focused ion beam (FIB). Several parameters were compared such as thickness, uniformity, preservation of original structure and composition.

Classical ultra-microtome is often used for soft materials. Before the sectioning the material needs to be fixed and embedded in an epoxy. No changes to the morphology were noticed during these steps. In spite of the retained composition and achievable thickness the classical ultra-microtome sections are often deformed during section resulting in a low success rate of an intact interface between the corrosion particles and the epoxy (fig1).

With FIB it is possible to directly sample with high selectivity the historical photograph. This is a great advantage when working with historical material where sampling is often restricted. Although it is possible to mill different materials several disturbing features are observed. FIB can cause preferential milling if the difference between the hard particles and soft matrix is big (see fig2). Also the low stiffness of the gelatine results in buckling during the thinning phase. These artefacts make it difficult to make a uniform TEM lamella, which is thin enough for analytical characterization. Any Ga+ implantation during preparation does not influence or disturb the characterization since Ga can easily be distinguished from the corrosion elements (fig 2C).

Since both techniques show artefacts making it difficult to achieve an intact thin and uniform sample a novel adaptation is suggested. Here we use the preparation steps of the classical ultra-microtome with an alternative final sectioning with focused ion beam. The difference between the classical ultra-microtome and ultra-microtome followed by FIB is the last stage or sectioning. This technique produces a TEM lamella with a clear interface and which is thin enough to determine the chemical composition or distribution of the nanoparticles in the corrosion layer (fig3). Although the success rate of this combined procedure is markedly better than that of the two alternatives, the main challenge remains making a thin enough sample to perform analytical characterization.

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Fig. 1: Artefacts with ultra-microtome; A: Overlapping of corrosion and image particles, B: intensity profile from image A showing large variation in deformation, C: successful interface corrosion particles and epoxy.

Fig. 2: Artefacts of FIB: A: buckling and preferential milling; B: curtain artefact in STEM image C: EDX spectra of Ga+ implantation (top) and intensity profile of marked area from figure B (Bottom).

Fig. 3: Combination of ultra-microtome and FIB; A: STEM overview, B: intensity profile of A, C: intact interface between corrosion particles and epoxy.