NANO- AND MICROTESTING OF HISTORICAL NOBLE METAL THREADS

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Besides wearing gold jewels man has woven noble metal threads into textiles and used them for embroidering his clothes for thousands of years. The first written record on the use of golden metal threads comes, as far as we know, from the Old Testament (Exod.39:2–3). Obtaining information on the manufacturing technique of textile decorating metal threads can help us to date and define the provenance of textiles and provides data also on the history of technology (JÁRÓ, 2010).

Solid gold (or gold looking) metal threads are basically of two morphological types (strips and wires). Both types were applied "bare" or wound around a fibrous core, in most cases silk yarn. In the beginning they were made of pure gold or gold alloys, but later on gilt silver, gilt-silvered copper and other metal combinations were also used (JÁRÓ, 1990). In order to reconstruct the manufacturing techniques (e.g., the way of gilding) of these composite threads, it is important to determine their fine texture and the related chemical composition. However, due to the limited dimensions of the threads themselves (STRIPS: width 150–1500 μm, thickness 7–40 μm; WIRES: diameter 70–500 μm, typically) and to the extreme thinness of the coatings (dominantly 10–300 nm), it is difficult to prepare a proper cross-section by traditional cutting and polishing, while in most cases simple surface analysis is not sufficient (e.g., ENGUITA *et al*., 2002; HACKE *et al*., 2004).

We prepared cross-sections of a dozen of 14th–17th century gilt silver threads by focused ion beam (FIB) milling. These cross-sections, as well as the surface of the threads, were studied by a combination of several techniques (high-resolution SEM, EPMA, EBSD, micro-Raman). Nanotexture and nanoscale chemical composition of the silver base metal, morphology of the surface gold coating, geometry of the gold/silver interface, as well as the corrosion features of the threads were examined.

The *silver base metal* of the examined metal threads contains copper both in solid solution and in dispersed particles. Micrometer and sub-micrometer sized solid inclusions inherited from the silver base metal preparation could also be identified. These observations help the reconstruction of the original metallurgical processes.

The thickness of the *gold coating*s was directly measurable (maximum thickness: 395 ± 5 nm). Finger-like intrusions of gold into the silver base metal are characteristic for the drawn wires, while in samples where subsequent manufacturing had a strong vertical component (post gilding hammering, rolling) smooth or wavy gold/silver boundary line can be seen.

At the *gold/silver interface* no enrichment of elements indicating special gilding techniques was found (Cu: use of a copper-based soldering material, Hg: fire gilding).

Most of the samples are affected by *corrosion*. Corrosion progressed along the grain boundaries and resulted in depletion of copper and formation of pores. Inside the pores corrosion products (silver chloride, silver sulphide) could deposit.

Our data indicates that the examined historical metal threads were presumably gilt by welding (not by fire gilding or soldering). The phenomenon that strips are of elevated copper content compared to drawn wires might reflect intentional technological choice: the use of copper-enriched silver base metal rather than copper-based soldering material (see also JÁRÓ, 2010).

The strong nano- and microscale textural inhomogeneity demonstrated here shows FIB/SEM as an inevitable, routinely used technique when characterizing historical metal threads and their manufacturing techniques.

The European Union and the European Social Fund have provided financial support to the project under the grant agreements no. TÁMOP 4.2.1./B-09/KMR-2010-0003 and TÁMOP-4.2.1.B-10/2/KONV-2010-0001.

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