# METAL THREADS MADE OF PROTEINACEOUS SUBSTRATES EXAMINED BY SCANNING ELECTRON MICROSCOPY—ENERGY DISPERSIVE X-RAY SPECTROMETRY

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Abstract—Scanning electron microscopy and energy dispersive X-ray spectrometry (SEM-EDS) have been employed along with the light microscope to describe some metal threads made of proteinaceous substrates taken from museum textile collections. The textiles are said to originate in Central Asia, Western Asia, and Syria or Egypt, thirteenth/fifteenth century AD. This study extends the set of data obtained in an earlier study of threads from the same time-period. A general methodology for the examination of these types of metal threads is proposed.

#### 1 Introduction

A number of studies of metal threads taken from historic textiles has been reported using modern analytical techniques [1-11]. The combination of light microscope and scanning electron microscope (SEM), equipped with energy dispersive X-ray spectrometry (EDS), has been useful for the description. In general, most structural details can be determined by light microscopy with the aid of a curator or conservator experienced in the examination of this type of material. A probe technique, e.g. X-ray fluorescence (XRF), particle induced X-ray emission (PIXE), energy dispersive X-ray spectrometry (EDS), capable of elemental analysis is also significantly useful [1-11]. In some recent studies, difficulties and pitfalls encountered in analyses have been enumerated [9, 10]. A general and important caveat applicable to all reporting and description is that the sample observed may not necessarily be representative of the textile as a whole:

- the sample specimen may come from a repair or later addition;
- the use of more than one technique within the same textile is entirely possible—a single textile may have both gold and silver threads, S and Z twisted wrapping, or both woven flat strips and wrapped strips;

Received 4 October 1988

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- 3) the quality of the details within the textile may show variations because of wear, aging, deterioration or workshop irregularities;
- 4) in the handling of the sample specimen structural details may be altered or lost, e.g. twists of core fibers or wrapping, looseness/tightness of wrapping.

It is very important to examine, visually and microscopically, the textile as a whole as well as the minute sample that is usually sacrificed for microscopic and/or elemental analysis.

Table 1 The five categories

- Metal applied (with adhesive) to already woven fabrics.
- II Metal wire or flattened strips.
- III Metal wire or strips wound around fiber core.
- IV Metallic surface applied (with adhesive) to organic wrapping wound around fiber core.
  - a. Organic = cellulosic.
  - **b.** Organic = proteinaceous.
- V Metallic surface applied (with adhesive) to organic strips.
  - a. Organic = cellulosic.
  - **b.** Organic = proteinaceous.

Five categories have been identified for the description of the use of metal in textiles (Table 1). The first category (I) identifies processes by which metal is applied with adhesive to the already formed textile without incorporating the metal into the primary structure of the fabric. The other categories indicate processes in which threads are made of metal without a core fiber (II); or with a core fiber (III); or metal is applied with adhesive to organic material (IV and V), twisted about a core fiber (IV) or not (V) and then incorporated into the textile either during the weaving process or as embroidered embellishment. Categories IV and V are subdivided according to whether the organic material is cellulosic or proteinaceous.

Table 2 Textiles from which samples were taken\*

Acc. no.	Museum	Catalog description
CMA 50.507	Cleveland Museum of Art	Central Asia, 13th-14th C
CMA 85.33	Cleveland Museum of Art	Central Asia, 13th-14th C
D 633	Copenhagen National Museum	Transoxiana (?), 12th-13th C
D 633 patch (cotton core)	Copenhagen National Museum	Transoxiana (?), 12th-13th C
D 633 patch (strip)	Copenhagen National Museum	Transoxiana (?), 12th-13th C
D 13	Brandenburg	Central Asia, 13th-14th C
T 883	Vienna: Mus. für angew. Kunst	Central Asia, 14th C
05905	Krefeld	Central Asia, 14th C
CMA 19.1004	Cleveland Museum of Art	Egypt/Syria, 14th C

<sup>\*</sup>These textiles have been described in greater detail in ref. 15. See Figure 1.



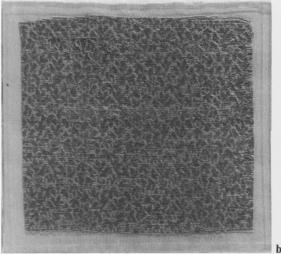


Figure 1a Silk and gold textile. Lampas weave, silk and gold thread. Cleveland Museum of Art, John L. Severance Fund, 50.507.

Figure 1b Silk and gold textile. Tabby weave, silk, with supplementary gold weft. Cleveland Museum of Art, Dudley P. Allen Fund, 85.33.

A recent study has attempted to categorize metal threads based on the analysis of a collection of textiles in the Indianapolis Museum of Art [6, 7]. The categories are defined according to the major metallic elements present. Important studies by Darrah [8] and Jaro [11] describing metal thread categories emphasize the method of manufacture. Others have also attempted to describe categories [1, 12–14], but with less sophisticated or less inclusive

definitions. In the present study only examples from categories IVb and Vb are examined.

# 2 Experimental

Examples were obtained from museum textiles (see Table 2 and Figure 1). The samples examined were taken from the specimens submitted. In each case, the organic strips and wrapping were arranged on spectroscopically pure carbon

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Figure 1c Fragment from a cope (detail). Lampas weave, silk and gold thread. Copenhagen, National-museet, D633A.

Figure 1d Patch sewn to cope (c). Compound twill weave, silk and gold thread. Copenhagen, National-museet, D633 fragment A; photograph: John Lee.

Figure 1e Silk and gold textile. Tabby weave, silk with supplementary gold weft. Brandenburg, Domstift, D-13.

stubs in a manner which would permit viewing (either with optical microscopy or scanning electron microscopy) of:

- 1) outer metal surface, metal clearly evident
- 2) outer metal surface, worn area
- 3) underside of organic material
- 4) core fiber

After being observed through a light microscope (Wild M8), the samples were carbon coated according to procedures already described [9, 10] and submitted to SEM-EDS analysis. Photomicrographs (see Figure 2) and printouts of EDS scans were retained for files.





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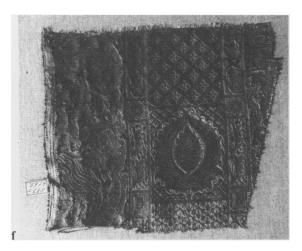
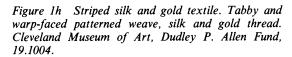
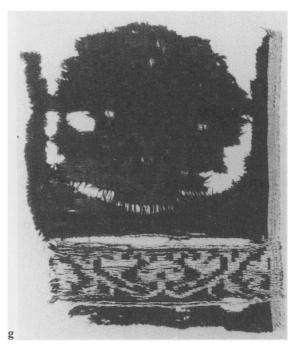
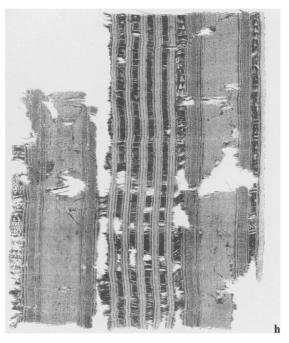


Figure 1f Silk and gold textile. Lampas weave, silk and gold thread. Vienna, Österreichisches Museum für angewandte Kunst, T883.

Figure 1g Fragment from a chasuble. Lampas weave, brocaded, silk and gold thread. Krefeld, Deutsches Textilmuseum, 05905.







#### 3 Results

Table 3 gives a general description of the specimens. Table 4 provides qualitative EDS analyses of elements observed of atomic number ≥11. Since all the metallic surfaces were, to some extent, abraded, elements present in the substrate and adhesive materials were detected in addition to the gold and silver. Table 5 is a summary of the results obtained including silver/gold ratios.

The results are generally similar to results obtained in earlier work [9, 10]. All the EDS analyses of the metallic surfaces of the proteinaceous substrates indicated a nearly pure gold surface consistent with analyses obtained for other textiles said to have been produced in Asiatic workshops [10]. D 633 was the lone counter-example: it is an ecclesiastical vestment, most of which is embellished with metal threads consisting of proteinacious strips with metallic surfaces (category Vb). The vestment contains an old repair patch (of similar date?), presumably inserted at a later date, which has in it both wrapped metal threads (D 633 patch cotton core, category IVb) and flat woven strips (D 633

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Table 3 Samples examined (light microscope and SEM images)

Acc. no.	Description of specimens
CMA 50.507  A B	Dark brown organic strip, brown in cross-section. Gold (leaf) on one side. Remnants of brownish silk warp perpendicular to strip on obverse. Leather. Outer surface. Metallic area. Inner surface
CMA 85.33 A B	Mottled yellowish gray organic strip. Unbroken gold surface on one side. Leather or parchment. Outer surface. Metallic area. Inner surface.
D 633  A B	Organic strip with silvery-gilt metallic surface on one side; much loss, dark adhesive visible. Rearside black with areas of translucency, dark adhesive visible. Membrane. Outer surface. Metallic area. Inner surface.
D 633 (patch, cotton core)  A B	Single wrapped undyed Z twist cotton core. Corroded organic wrapping Z twist. Very little metal left, dark adhesive, areas of translucency? Membrane or leather. Wrapping, outer surface, metallic area. Core fibers.
D 633 (patch, strip)  A B	Organic strip. Mottle gilding (leaf not apparent) on one side, adhesive, whitish substrate, adhesive appears to be on rearside. Membrane.  Outer surface. Metallic area.  Outer surface. Nonmetallic area.
D 13 A B	Organic strip. Bright gold (leaf) with cracks of white showing through on one side; mottled light brown on underside. Parchment or vellum. Outer surface. Metallic area. Inner surface.
T 883 A B	Dark brown organic strip, brown in cross-section. Unbroken gold (leaf) on one side. Leather. Outer surface. Metallic area. Inner surface.
Krefeld 05905	Core: single wrapped cotton, slight Z twist, white, undyed. Wrapping: gold on dark brown leather, Z twist, evenly wrapped. More gold (wrapping) showing than core— $c.2$ –3:1 wrapping:core. Substrate not translucent. Metal.
B C	Substrate. Core fibers.
CMA 19.1004 A	Core: single wrapped, untwisted, very white. Wrapping: gold on brown leather? 5 twist, brown cross-section, possible regions of translucency (membrane?)  Metal.
В	Substrate. Core fibers.
C D	Core fibers.



OLCMA 19.1004 01KV X 70 100U 108 615U MMA

Figure 2a SEM photomicrograph of specimen from Figure 1g.

Figure 2b SEM photomicrograph of specimen from Figure 1h.

Table 4 EDS results

Acc. no.	Elements observed*	
CMA 50.507	A B	Gold ( $\sim$ 75%), silicon, calcium, silver ( $\sim$ 4.5%), aluminum, chlorine, potassium, iron, other trace elements. Ag/Au: $6 \pm 2/94 \pm 2$ . Calcium, silicon, chlorine, potassium, aluminum, iron, sodium, sulfur, magnesium, other trace elements
CMA 85.33	A	Gold, no silver, silicon, calcium, aluminum, potassium, chlorine, iron, other trace elements. Ag/Au: 0/100.
D 633	A	Silver ( $\sim$ 46%), gold ( $\sim$ 30%), calcium, silicon, potassium, iron, chlorine, aluminum, other trace elements. Ag/Au: $60 \pm 3/40 \pm 3$ .
D 633 (patch cotton core)	A B	Silver ( $\sim$ 46%), gold ( $\sim$ 30%), silicon, calcium, aluminum, iron, chlorine, other trace elements. Ag/Au: $60 \pm 3/40 \pm 3$ . Silicon, calcium, aluminum, silver, iron, sulfur, phosphorus, potassium, other trace elements.
D 633 (patch strip)	A B	Silver ( $\sim$ 48%), gold ( $\sim$ 34%), calcium, silicon, chlorine, aluminum, iron, other trace elements. Ag/Au: $60 \pm 3/40 \pm 3$ . Calcium, silicon, sulfur, aluminum, magnesium, iron, other trace elements.
D13	A	Gold ( $\sim$ 87%), no silver, silicon, calcium, potassium, iron, other trace elements. Ag/Au:0/100.
T 883	A	Gold ( $\sim$ 85%), silicon, silver ( $\sim$ 4%), calcium, potassium, iron, other trace elements. Ag/Au: $4 \pm 3/96 \pm 3$ .
Krefeld 05905	A B C	Gold (~95%), silver (~3%), copper, calcium, traces. Calcium, iron, silicon, sulfur, copper, other trace elements. Calcium, copper, potassium, silicon, iron.
CMA 19.1004	A B C, D	Gold (>95%), copper, other trace elements, no silver. Calcium, silicon, iron, copper, gold. Calcium, silicon, iron, copper, gold, other trace elements.

<sup>\*</sup>Elements of atomic number  $\geq 11$ .

Metal threads made of proteinaceous substrates examined by SEM-EDS

Table 5 Summary of findings

Acc. no.	Description of specimens
CMA 50.507	Dark brown organic strip, brown cross-section. Gold (leaf) on one side. Leather. Width of strip: ~510μ (0·51mm). Ag/Au:6 ± 2/94 ± 2.
CMA 85.33	Mottled yellowish gray organic strip. Unbroken gold surface. Leather or parchment. Width of strip: $\sim 460 \mu$ (0·46mm). No silver detected.
D 633	Organic strip with silvery-gilt metallic surface; much loss of metal, dark adhesive. Rearside black (adhesive?) with areas of translucency. Membrane. Width of strip: $\sim 545 \mu$ (0.545mm). Ag/Au: $60 \pm 3/40 \pm 3$ .
D 633 (patch cotton core)	Single wrapped undyed Z twist cotton core. Corroded organic wrapping Z twist. Very little metal left, dark adhesive, areas of translucency (?) Membrane or leather. Width of wrapping: $\sim 565 \mu$ (0·565mm). Thickness of wrapping: $\sim 27 \mu$ (0·027mm). Diameter of core fiber: $\sim 750 \mu$ (0·75mm). Ag/Au: $60 \pm 3/40 \pm 3$ .
D 633 (patch strip)	Organic strip. Mottled gilding (leaf not apparent), brownish adhesive, whitish substrate, adhesive appears to be on rearside. Membrane. Width of strip: $\sim 660 \mu$ (0.66mm). Ag/Au : $60 \pm 3/40 \pm 3$ .
D 13	Organic strip. Bright gold (leaf) with cracks of white showing through; mottled light brown on underside. Parchment or vellum. Width of strip: $\sim 570\mu$ (0·57mm). No silver detected.
T 883	Dark brown organic strip, brown in cross-section. Unbroken gold (leaf). Leather. Width of strip: $\sim 555 \mu$ (0.555mm). Ag/Au: $4 \pm 3/96 \pm 3$ .
Krefeld 05905	Single wrapped light yellow fiber (cotton), Z twist. Z twist wrapping, loosely wrapped leather to which is attached gold; traces of silver and copper. Width of wrapping: $\sim 970 \mu$ (0.970mm).
CMA 19.1004	Single wrapped undyed fibers (silk, S plyed). S twist wrapping of brown membrane to which is attached gold metal (trace of silver). Width of wrapping: $\sim 615 \mu$ (0·615mm).

patch strips, category Vb). The EDS analyses of all three specimen surfaces taken from D 633 gave similar elemental analyses for the gold and silver compositions, and these analyses are consistent with analyses obtained for textiles woven in Spain or Italy during the same time-period [10]. Numerous technical and stylistic features of the textile suggest an Asiatic origin [15]. The elemental analyses are consistent with analyses of thread taken from textiles manufactured in some Mediterranean center rather than in an Asiatic center. It is entirely possible that there is a group of Asiatic textiles with similar elemental composition to these.

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### 4 Discussion

## 4.1 Category IV

Category IV comprises metallic surface applied (with adhesive) to organic wrapping wound around fiber core; metallic surface on leather, parchment (vellum), membrane, paper, wrapped around fiber core.

Many textiles of the Middle Ages contain threads that belong to this category. In recent studies [9, 10, 14–16] of specimens from mediaeval Italian, Spanish, Western Asiatic and Mamluk textiles, the metal composition observed on thread samples (especially gold/

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silver ratios) appeared to be a possible indicator of geographic origin.

Among examples of this category studied to date only leaf (c.  $1-5\mu$  thick) has been observed. Occasionally the overlapping that occurs from the application of adjacent leaves has been noted [9]. The metallic surface adheres to the wrapping material through an adhesive; the wrapping material adheres to the core fiber through the twist of the wrapping material about the core fiber. Between the wrapping material and the core fiber no layer of adhesive is used to make the core fibers adhere to the substrate, but sometimes adhesive may be detected on the underside of the substrate, and often there is evidence of migration of aged adhesive and wrapping material into the core fibers. No systematic studies have appeared in the literature that have attempted to identify the adhesives used for the manufacture of category IV or V threads. The adhesives are likely to be related to those described for the application of leaf to early manuscript and bookbinding illumination, e.g. bole, gum, gelatin, glue, egg white, etc.

Aside from the general difficulties in distinguishing among cotton/linen/silk for deteriorated (aged) samples, the identification of the wrapping material can be significantly hindered by the decomposition of the material itself [9, 10]. Cross-sectioned samples from CMA 50.507, CMA 85.33, D 13 and T 883 have been examined in the H. F. du Pont Winterthur Conservation Laboratory and the substrates identified as tanned vellum or skin [17].

Parchment [18] is made from animal skins: goat, sheep, deer, steer, camel, etc. The skin is stretched, salted to remove hair follicles, usually whitened with chalk or clay, and treated and abraded in a variety of ways depending on the desired end use. Vellum is a term often used interchangeably with parchment, but it is sometimes used to refer specifically to material made from young or unborn animals or to skins of high quality. An unfortunate terminological ambiguity arises from the modern practice of naming some treated papers 'vellum' or 'parchment'. The principal proteinaceous constituent of parchment or vellum is collagen. If the wrapping substrate material is very white (observed with a light microscope) it may be paper or parchment. Parchment tends to yellow but many old samples of parchment remain very white, even with considerable age. Some old samples of parchment have areas of translucency, possibly from handling or treatment during manufacture. Depressions left from depilated hair follicles are occasionally observable microscopically on vellum and parchment [18].

Specimens of leather from historic textiles in which the leather has been used as the wrapping substrate for the applied metal are brown in cross-section, lack areas of translucency and usually have brown to blackish adhesive material holding the metal to the leather [9, 10]. Similarly to parchment, depressions left from depilated hair follicles may be observed microscopically [18]. The absence of depressions is an unreliable indicator for either parchment (vellum) or leather, since any particular specimen may lack such depressions. Hair may be absent from a localized area or the refining process may have removed an epidermal layer thick enough to include the depressions. The proteinaceous constituent of leather, like parchment and membrane, is collagen. Leather differs from parchment and membrane in having undergone the process of tanning. The process serves to remove the animal hair, crosslink the protein chains, and color the material.

Membranous material [18, 19] (from the stomach or intestinal walls of lean cattle; occasionally from certain fish bladders) has extended areas of translucency ranging from colorless to light brown, often interrupted by black or dark brown decomposition products from the adhesive. The adhesive material holding the metallic surface to the membranous wrapper is said to be the natural exhudates associated with the membrane as obtained from the dead animal [20]. Historical literature often refers to the thread made from this material as Cyprian gold [12, 20-23]. The membranous material itself is sometimes identified as goldbeater's parchment or goldbeater's skin [18, 23], named according to its traditional use in separating gold leaf during the process of beating [19]. This material lacks hair follicle depressions.

The variation in raw materials (differences in the animals from which leathers, parchments or membranes are taken) and variations in the processing or manufacture of the metal thread may result in considerable uncertainty in the description of these materials. The substrates are also subject to decomposition, embrittlement, wear

Table 6 What to look for in Category IV specimens

- 2 Composition of metal surface, corrosion
- 3 Composition of adhesive layer between metallic surface and wrapping material
- 4 Identity of wrapping material
- 5 Thickness of core and wrapping and surface
- 6 Twist of core and wrapping
- 7 Looseness/tightness of wrapping (pitch)

and treatment damage, and often have a radically different appearance from the original product. The distinctions among paper, parchment, membrane and leather have, unfortunately, often been clouded in accounts of the use of these materials, as well as in the textile literature.

The amount of metal necessary to produce this type of metallic thread is obviously less than for category III, but the pliability of the wrapping material affords longevity and variety of

Table 7 Appearance (magnified) of organic wrapping materials found on historic textile threads. Category IV

Wrapping material	Chemical composition	Appearance	Comments
Paper IVa	Cellulose	White yellow brown. Opaque; no translucence. No hair follicle pits.	Adhesive material usually visible between metal surface and paper. Silk core.
Membrane IVb	Collagen	Colorless offwhite pale brown. Translucent areas. No hair follicle pits.	Adhesive material not uniformly visible. Dark areas of degraded adhesive. Silk, cotton or linen core.
Parchment IVb	Collagen	White yellow. Generally opaque; sometimes nearly translucent areas. Hair follicle pits.	Adhesive material visible. Dark areas of degraded adhesive. Silk, cotton or linen core.
Vellum IVb	Collagen	Same as parchment. Hair follicle pits may be sparser and smaller than in parchment.	Same as parchment.
Leather IVb	Collagen	Brown. Hair follicle pits larger than in parchment.	Same as parchment.

Table 8 Varying texture and color of wrapped metal threads, Category IV (wrapping = metal applied to organic substrate)

- a. Composition of metal (applied as leaf). Gold, silver, gilt silver, baser inclusions.
- b. Wrapping substance. Paper [IVa]. Membrane, parchment, vellum, leather [IVb].
- b'. Adhesive. Bole, glue, gum, etc.
- c. Geometry of the wrapping material. Thickness, width.
- d. Interaction of wrapping and core. Pitch of wrapping metal (exposure of core); color of core; plying of core; plying of wrapped thread with colored thread.
- e. Color of warp (in brocades) or couching threads (in embroidery).

texture and visual effect.

Table 6 outlines what to look for in category IV specimens. Table 7 summarizes the appearance under magnification  $(c. 10-50 \times)$  of the organic wrapping materials. Table 8 outlines features to observe (apart from weaving or

Table 9 What to look for in Category V specimens

- 1 Identity and fabric fibers
- 2 Composition of metal surface, corrosion
- 3 Composition of adhesive layer between metallic surface and strip
- 4 Identity of strip material
- 5 Thickness of strip and surface

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Substrate	Comments	Reference
Paper Va	Gold or silver applied to one side. Woven into silk or bast fiber fabrics. Embroidered. Appliquéd.	28 (Japanese) 29-32 (Indonesian) 24 (Central Asian)
Leather, membrane, parchment, vellum <b>Vb</b>	Gold or silver applied to one side. Woven into silk fabrics.	33, 34 (Chinese)

Table 10 Metal thread strips; organic substrates without core (Category V)

stitching techniques) of metal threads with a core fiber in which the wrapping material is an organic substrate.

4.2 Category V: metallic surface applied (with adhesive) to organic strips

Some textiles contain woven or embroidered elements consisting of thin paper or proteinaceous strips with metallic surfaces. Proteinaceous strips are found only in early Asiatic examples (sometimes identified as membrane [24]); paper is generally found on later Chinese and Japanese textiles. Examples of couched gilt paper are known on ninth/tenth century embroideries from Central Asia [25]. The metal is applied to only one surface of the strip and only the metallic surface appears facing the outside of the textile. The metal may be leaf with an adhesive that provides intonation to the metallic color or metallic paint. These features may occur in textiles that also contain other kinds of metallic threads to achieve textural and tonal variety. This category of metallic thread is similar to category II, flattened strips of metal, and is clearly cheaper to produce; it may also have greater longevity owing to the flexibility of the organic substrate. The use of threads from this category is sometimes referred to as eastern strapwork [26, 27]. In embroideries, material of

Table 11 Varying texture and color of metal thread strips; organic substrate without core (Category V)

this category is couched. Certain Indonesian textiles have paper appliqués that correspond to this category. A useful subcategory is defined according to the nature of the organic substrate (cellulosic, Va; proteinaceous, Vb).

Table 9 outlines features of category V specimens that may serve to characterize a specimen. Table 10 summarizes the types of metal thread which consist of metal applied with adhesive to an organic substrate with no core (category V). Table 11 outlines features (apart from weaving techniques or stitching processes) which may be observed among specimens of this category to account for varying color and texture.

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a. Composition of metal (applied as leaf). Gold, silver, gilt silver.

b. Substrate. Paper, leather

b. Adhesive. Bole, glue, gum, etc.

c. Geometry of the substrate. Thickness, width.

d. Color of warp (in brocades) or couching threads (in embroidery).

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Résumé—Le microscope électronique à balayage et la spectrométrie de rayons X en dispersion d'énergie, ainsi qu'un microscope à faible grossissement, ont été utilisés pour étudier certains fils métalliques à substrat protéineux provenant des collections textiles du musée. Ces textiles passent pour être originaires d'Asie Centrale, d'Asie occidentale, de Syrie ou d'Egypte et sont probablement datés du XIIIe/XVe S. de notre ère. Ces examens prolongent l'ensemble des resultats obtenus dans une précédente étude de fils de la même époque. On propose un essai de méthodologie générale pour l'examen de ce type de fils de métal.

Zusammenfassung—Unter Einsatz der Lichtmikroskopie, der Rasterelektronenmikroskopie und der energiedispersiven Röntgenfluoreszenzspektrometrie werden einige Metallfäden auf der Basis proteinhaltiger Träger beschrieben. Die untersuchten Proben entstammen musealen Textilsammlungen, Sollen aus Zentralasien, Westasien, Syrien oder Ägypten stammen und aus dem 13, bis 15 Jh. sein. Der Beitrag erweitert das Datenmaterial einer früheren Untersuchung gleichartiger Fäden derselben Datierung und schlägt eine generell gültige Vorgehensweise zur Untersuchung dieses Typs von Metallfäden vor.