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Crystallographic texture analysis: applications in mineralogy and archaeometry

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ABSTRACT. — Texture analysis by diffraction methods has greatly advanced in the last few years because of instrumental and computational developments, and it is now to be considered a routine tool for the analysis of crystallite orientation in a wide variety of materials, including rocks, industrial products, and archaeological samples. The advances in the experimental measurements are mainly linked to the use of flexible experimental setups at large radiation sources, such as synchrotrons and neutron sources, which allow faster data collection, the use of samples of any size, and complete coverage of texture and reciprocal space. The developments in the data analysis are mainly related to the use of the full diffraction profiles in place of the single-peak methods. This produces pole figures and orientation distribution functions (ODF) that are statistically more significant and less prone to biases in the data analysis. Furthermore it opens the possibility of analysis of complex polyphasic materials, which are hard to characterize by other experimental techniques. Applications are discussed concerning the texture analysis of metamorphic rocks and archaeological samples.

RIASSUNTO. — L'analisi tessiturale basata su misure diffrattometriche ha avuto negli ultimi anni un grande sviluppo dovuto ad avanzamenti sia strumentali che computazionali, tanto che si può ora considerare uno strumento diffuso di analisi dell'orientazione dei cristalliti in un ampio spettro di materiali, inclusi rocce, prodotti industriali e campioni archeologici. Gli sviluppi nelle misure sperimentali sono soprattutto legati all'utilizzo di configurazioni di misura flessibili presso le grandi sorgenti di radiazione, quali sincrotroni e sorgenti di neutroni, le quali permettono raccolte dati più veloci, la misura di campioni di qualsiasi taglia, e la copertura completa dello spazio tessiturale e dello spazio reciproco. Gli sviluppi nell'analisi dati sono principalmente legati all'impiego dei metodi di raffinamento a profilo completo al posto di quelli basati su singoli picchi di diffrazione. Tutto questo permette di ottenere figure polari e funzioni di distribuzione dell'orientazione (ODF) statisticamente più significative e meno soggette ad errori sperimentali. Inoltre il trattamento a profilo completo permette di affrontare l'analisi tessiturale di materiali polifasici complessi che sono di difficile caratterizzazione mediante altre tecniche analitiche. Sono qui discussi esempi di applicazioni che riguardano l'analisi tessiturale di rocce metamorfiche e campioni archeologici.

KEY WORDS: texture analysis, Rietveld refinement, neutron diffraction, archaeometry, rock fabric

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INTRODUCTION

The texture analysis of polycrystalline materials is important for the interpretation of the physical and mechanical properties of natural and synthetic crystalline materials. The atomic structure, the size, the shape, and the orientation distribution of the crystal domains in solids control the measurable tensor properties such as the electrical conductivity, the thermal expansion, the compressive strength, the resistance to corrosion, or the elasticity, among many other. In the Earth Sciences for example, the determination of the orientation distribution of crystallites in rocks is the starting point to model the transmission of seismic waves or the conductivity in the lithosphere, or to understand how rock formations undergo plastic deformations and/or recrystallization by diagenesis, metamorphism, or other geological processes. In archaeometry, texture analysis yields information for the interpretation of the manufacturing techniques of archaeological and art objects of different nature, especially ceramics and metal tools. The ability to properly describe the crystal texture in samples having a strong preferred orientation is also essential when performing quantitative phase analysis, for example in the characterization of raw industrial minerals and products.

Geological and industrial materials are generally complex polymineralic solids and they rarely consist of a single phase. Therefore the application of diffraction texture analysis procedures based on single peak methods are hardly conclusive, mainly because of severe peak overlap and absorption problems. In the last few years, the use of diffraction texture analysis based on full profile methods has developed considerably, and it may represents a valid procedure for the texture analysis of complex materials. Extension of the methods by the use of penetrating beams such as high energy X-rays and neutrons make the technique a powerful tool for the non-destructive analysis of valuable archaeological and art objects.

CRYSTALLITE ORIENTATION DISTRIBUTION AND SAMPLE TEXTURE

The determination of the crystallographic texture in a polycrystalline sample can be rationalized by describing how the crystallographic coordinate system K_B, relative to each crystallite (i.e. each coherently diffracting crystal domain), is oriented with respect to the reference coordinate system K_A , relative to the investigated sample. The problem is analogous to the Eulerian change of reference frame of a vector triplet, that is we need to define the set of rotations (g: $[K_A \rightarrow$ $(K_{\rm R})$ to be applied to bring one frame $(K_{\rm A})$ parallel to the other (K_B), where the symbol g = $g_1 g_2 g_3 = \{\alpha, \beta, \gamma\}$ denotes the set of three Eulerian angles according to Matthies' convention (Matthies et al., 1987):

- rotation of K_A about the axis Z_A through the angle α : $[K_A \rightarrow K'_A]$; $g_1 = \{\alpha, 0, 0\}$

- rotation of K'_A about the axis Y'_A through the angle β : [K'_A \rightarrow K''_A]; g₂ = {0, β , 0}

- rotation of K''_A about the axis Z''_A through the angle γ : [K''_A \rightarrow K'''_A || K_B]; g₃ = {0, 0, γ }

Obviously, the inverse rotation is also possible, defined as g^{-1} : $[K_B \rightarrow K_A]$. The space containing all possible orientations of K_B relative to K_A (and vice versa) is called the orientation-space or G-space. G is a finite space, as $0 \le \alpha$, $\gamma \le 2\pi$ and $0 \le \beta \le \pi$, with its infinitesimal element being dg = $d\alpha \sin\beta d\beta d\gamma$ and having the volume

$$\int_{G} dg = \int_{G} \sin \beta d \beta d \alpha d\gamma = \int_{0}^{2\pi} d \alpha \int_{0}^{2\pi} d\gamma \int_{0}^{\pi} \sin \beta d \beta$$
$$= 8\pi^{2}.$$

The texture of the sample can then be easily described by a discrete number of orientations, i.e. definite intensity values of occupation density in a set of contiguous (but finite) cells of the G-space or, similarly, by a distribution function f(g) of the orientations g which is

known as ODF (Orientation Distribution Function). The G-space is commonly represented by a rectangular prism in Cartesian coordinates and by its 2-dimensional y sections or, alternatively, by an equal area projection (Matthies et al., 1987; Kocks et al., 1998). In practice, the complete orientation distribution functions describes how many crystallites in the probed volume of the sample possess an orientation g. Due to its statistical meaning, the ODF is commonly normalized as 1 $\frac{1}{8\pi^2}\int f(g)dg = 1$, and a relative unit is used so that $f(g)_{random} = 1$ m.r.d (multiple of random distribution), negative values (f(g) < 0) being therefore meaningless.

Orientation distributions are usually represented as pole figures, i.e. 2D density plots of the ODF, showing the probability of a crystal direction **h** (defined in the $K_{\rm B}$ system) to occurr parallel to an arbitrary sample direction \mathbf{y} (defined in the K_A system). Pole figures represent the density of crystal poles as a continuous function of the polar (θ) and azimuthal (φ) angles. Kallend (in: Kocks *et al.*, 1998) shows as an example that if y is the direction of the scattering vector s of a diffractometer (in symmetric parafocusing geometry) relative to the sample coordinate system and **h** the normal to (00l) lattice planes of an orthorhombic phase, when the Bragg condition for the (00l) planes is satisfied, the distribution ($\mathbf{f}(\theta, \varphi)$) of the intensities diffracted by the sample is like a pole figure. Moreover, since the Bragg condition is always satisfied upon rotation of every crystallite around its pole (001), we can have a direct example of angular relation between the two reference frames K_A and K_B , as $\beta = \theta$, $\alpha = \varphi$, while γ may have any value. Then, the pole figure of an arbitrary (hkl) can be obtained integrating the ODF along а path corresponding to a 2π rotation of every crystal about its (*hkl*) pole. The relation becomes:

$$P_{(hkl)}(\theta, \varphi) = \mathbf{P_h}(\mathbf{y}) = \frac{1}{2\pi} \int_0^{2\pi} f(g) \, d \, \psi$$

where ψ is a rotation angle about the common direction $\mathbf{h} \parallel \mathbf{y}$. It is thus evident that pole figures are 2-dimensional projections of the 3-dimensional ODF in the sample space. According to their statistical meaning, pole figures are also normalized such that $\int_{y} P_{\mathbf{h}}(\mathbf{y}) d\mathbf{y} = \int_{0}^{\pi} \sin \theta d\vartheta \int_{0}^{\pi} d\varphi P_{h}(\mathbf{y}) = 4\pi$ and $P_{(hkl)}(\theta, \varphi)_{random} = 1$ m.r.d. Also, an inverse pole figure $\mathbf{R}_{\mathbf{y}}(\mathbf{h})$ can be derived as being in crystal coordinate system the probability that a sample direction \mathbf{y} is parallel to an arbitrary crystal direction \mathbf{h} , i.e. the 2D projection of the OD in the crystal space.

EXPERIMENTAL DETERMINATION OF THE ODF

The direct experimental determination of the ODF may be performed using optical or electron microscopy. The measurement of the orientation of each crystallite by optical microscopy is rather laborious, it requires the use of a universal stage, it can only be performed on thin sample sections, and furthermore optical aberrations commonly limit the analysis to crystallites larger than few μ m. The electron microscopy techniques require a SEM equipped with a detector for the diffraction analysis of the backscattered electrons (EBSD – Prior and Lloyd, 2000). The technique is in principle rather powerful, as it allows direct interpretation of the orientation of the small volume of the crystallite probed by the electron beam. The sequential analysis of the diffraction images produced by the probed points in a raster scan yield the size, shape and orientation of each crystallite. However, the use of an electron beam limits the use of EBSD only to the surface of the sample, and furthermore the diffraction signal in lowsymmetry polyphasic samples can not yield unambiguous orientation information. The technique at present is therefore mostly limited to monophasic samples.

By far, the most widely used technique for the indirect determination of ODF involves the measurement of the 2-D pole figures by X-ray or neutron diffraction and the subsequent 3-D ODF recalculation using the so-called pole figures inversion method. However we ought to be aware that by using diffraction for the texture analysis, the recalculated orientation distribution has a certain degree of ambiguity. In fact only a reduced pole figure, $\tilde{\mathbf{P}}(\mathbf{h}_i)(\mathbf{v}) =$ $[P(h_i)(\mathbf{y})+P(-h_i)(\mathbf{y})]/2$ can be measured because of Friedel's law intrinsic to diffraction. Consequently a reduced ODF is also determined, sometimes leading to the appearance of ghost ODF peaks that have no corresponding components in the true texture. The main existing problems of texture measurements with X-ray and neutron diffraction techniques can then be summarized as follows: (1) the measurement of a limited number of pole figures, which provide only part of the information of the ODF, and (2) part of the OD information is lost during the pole figure inversion.

The described limitations may partially be overcome by applying physical or numerical constraints to the adopted inversion algorithms. Such additional constraints generally imply (Bunge, 2000): (1) the assumption that the ODF is positive in the orientation space, (2) the assumption that the background of the f(g) is high enough to hide likely ghost peaks, and (3) that possible solutions to be taken into account should have smooth peaks having well-defined Gaussian or Lorentzian shape. The ODF can then be computed either by a spherical harmonics approximation in the Fourier space (Bunge, 2000), or by iterative procedures in the direct G-space, such as the WIMV method (Matthies and Vinel, 1982) or the vector method (Ruer and Baro, 1977).

POLE FIGURE MEASUREMENTS

Diffractometers used for pole figure measurements must have a cradle allowing the sample to be rotated around four axes, and a detector to collect the diffracted radiation. In a diffraction experiment the condition $\mathbf{s} \parallel \mathbf{h}$ must be fulfilled to satisfy Bragg law and, in case of a polycrystalline sample the condition $\mathbf{s} \parallel \mathbf{y}$ is

also valid, depending on the orientation of the crystallites with respect to the sample direction y. As the diffractometer rotation angles are conventionally reported as θ , ω , χ , φ , here we prefer to define them the Euler angles. The azimuthal and polar angles of pole figures are α and β , respectively, and θ is the Bragg angle. If $\mathbf{y} = g \cdot \mathbf{s}$, and $g = \{\omega \chi \varphi\}$, two sample rotations (i.e. the conventional χ and φ angles) are sufficient to bring any sample direction parallel to the scattering vector at any 2θ position. The diffraction pattern of a textured sample can be considered the intermediate case between the diffraction pattern of a single crystal and that of an ideally random powder.

Pole figures data collection can be performed either with X-rays or neutrons, the former being preferred in the study of textured sample surfaces and thin layers, whereas the low absorption cross section of most materials for neutrons, make them more suitable for thick and large volume specimens. As texture experiments generally take several hours to guarantee a suitable coverage of the pole figure, area detectors are now generally preferred to conventional point detectors in order to reduce the measuring time.

Textures are conventionally reconstructed by measuring how the diffracted intensities vary with the tilting of the sample with respect to the incident radiation. The integrated intensities are extracted from a set of well-indexed reflections, and the ODF is computed. This classical procedure can often be successfully applied to monophasic samples, especially when the crystal phases have a high crystallographic symmetry and therefore the diffraction pattern shows well resolved peaks, but it is not generally applicable to rocks or other geologic samples, because they are normally polyphasic and their powder diffraction patterns show a high degree of peak overlap. Furthermore they are often rather heterogeneous in crystal size, shape and absorption contrast, which make the classical approach useless in most cases. The recently introduced technique presented by Lutterotti et al. (1997) extracts the integrated intensities of

all peaks in the diffraction pattern via a Rietveld-type simulation and refinement of the full intensity profile. The so-called RIetveld Texture Analysis (RITA) method has proven to be a powerful tool to overcome the peak overlap problem and to resolve the textures of complex geological samples (Wenk et al., 2001; Wenk, 2002). Furthermore, the added possibility to simultaneously treat the peak shifts due to strong residual stresses (i.e. the RIetveld Stress Texture Analysis or RISTA method) extends the technique also to the case of samples that underwent large plastic deformations, such as in minerals deformed under extreme non-hydrostatic pressure conditions. Here we present a novel successful application of the RITA method to rock and archaeological samples.

EXAMPLE 1:

TEXTURE ANALYSIS OF AMPHIBOLITIC ROCKS

The sample studied is an amphibolite rock, mainly constituted by amphiboles of winchiterichterite composition (~95 wt%), and minor amounts of chlorite, rutile, quartz, and zircon. The specimen is peculiar of the Eclogitic Micaschists Complex (EMC) of the Sesia-Lanzo zone, Western Alps, Italy, which is a well known case area for the study and the interpretation of the high grade metamorphic processes acting during tectonic events. Diffraction analysis of textural features of the minerals ought to help quantifying the overall deformation mechanisms of the rock. The selected sample is characterized by a marked mineral iso-orientation, clearly visible by crossed polarization optical microscopy, which is parallel to the main rock foliation and it is defined by two generations of amphiboles showing a bimodal grain size distribution. A representative sample of the amphibolite rock was initially powdered in an agate mortar and a powder pattern was collected using Cu Ka radiation, angular range 5-70 °20, 0.01 °20 steps, and counting time 3 s/step. Preliminary qualitative and quantitative phase analysis revealed the presence of chlorite as a minor phase (~5 wt%), the other phases observed by optical microscopy (rutile, quartz, and zircon) being below the detection limits of diffraction (estimated at about 0.1 wt%). The quantitative phase analysis was performed by the fullprofile Rietveld method using the richterite and the clinochlore structural models for the two mineral phases. The ideal richterite structure model taken from the literature was then modified during the refinement to take into account the crystal chemistry of the sample measured by EPMA. The adopted clinochlore structure model, although probably inadequate to represent the sample chemistry as shown from the refined cell parameters and the poor cristallinity of this phase, formerly evidenced in thin section microscopy, was deemed sufficient to model the chlorite diffraction peaks and extract the textural information, because of the low amount of the phase.

A 1.0 cm³ specimen was then cut from the rock sample and mounted on the texture cradle available at the D1B beamline of the ILL neutron reactor, Grenoble, France. The use of an area detector simultaneously covering an angular range of 80 °2 θ allowed collection of 1368 powder patterns, corresponding to a coverage of the sample space of 0-360° in ϕ and 0-90° in χ , using 5° steps and a wavelength of 2.54 Å. The collected patterns have been processed with the program package MAUD (Lutterotti et al., 1999), which can perform a combined Rietveld-Texture analysis (RITA method) simultaneously on a large number of powder profiles (most other available Rietveld programs are limited to a few tens of profiles and they are not specifically designed for texture analysis). As a first step the texture was assumed to be random, and the following parameters were refined on the average sum profile: cell parameters, peak shape function, background function and phase scale factors. Subsequently, the previously obtained instrumental broadening and χ -dependent background functions were used in the simultaneous refinement of all 1368 profiles, combined with the texture analysis of the amphibole phase. The ratios between the



Fig. 1 – Three patterns refined without texture is computed (a) and after WIMV processing (b). Discrepancies due to texture are clearly visible for certain reflections such as the 0/0. Intensities in arbitrary units.

Refined lattice parameters and phase relative amount.									
Phase	a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)	weight %		
Amphibole Chlorite	9.53558(8) 5.0646(2)	17.7091(2) 8.3633(3)	5.28237(7) 16.9841(8)	90 90.635(4)	103.7801(8) 97.661(4)	90 81.668(3)	96.2 4.8		

 TABLE 1

 Refined lattice parameters and phase relative amount

experimentally measured intensities of the peaks and the theoretical ones for a random crystallite distribution are determined with the Le Bail algorithm (Le Bail et al., 1988), and the difference is attributed to texture effects. The resulting experimental pole figures are then used for the recalculation of the ODF by the WIMV method. The iterative procedure is repeated until a minimum in the least squares refinement of both Rietveld and WIMV is obtained. Figure 1 shows three fitted powder profiles taken at different angles before and after the texture correction: discrepancies attributed to texture are clearly visible. especially on the basal and prismatic planes of both mineral phases. Refined lattice parameters are reported in Table 1, statistical texture data and residues reported in table 2 correspond to the experimental pole figures shown in fig. 2.

The agreement between the experimental normalized pole figures and the recalculated ones is excellent (fig. 2), thus suggesting that the calculated ODF extrapolation is correct. As can be seen in Table 1, the texture index ($F^2 =$ 6.33) indicates a strong texture of the amphibole crystals. The plotted pole figures in fig. 2 are relative to the principal crystallographic directions. They show that the [001] poles are located at an angle of $\sim 10^{\circ}$ with respect to the main mineral lineation observed by optical microscopy. The [010] and [100] poles are essentially dispersed in the plane perpendicular to the lineation direction. The observed texture is compatible with an hypothetical rock deformation based on an active slip system on (100)[001] (that is a [001] slip on the (100) plane), as expected from the main cleavage planes derived from the amphibole crystal structure.

TABLE 2

Texture data, refinement residues, and statistical texture errors for amphibole (for terminology please refer to Matthies et al., 1988).

N. of pole figures used	R _w	RP ₁ (%)	F ² (m.r.d. ²)
17	0.1053	10.74	6.33

EXAMPLE 2:

METAL TEXTURES OF PREHISTORIC COPPER AXES

For average absorbing materials, texture analysis performed with conventional X-rays gives information only on the surface layer of the sample, whereas the same analysis performed with deeply penetrating neutrons yields a more appropriate interpretation of the ODF in the bulk. Furthermore neutrons are totally non-destructive probes of the matter, as they interact with atomic nuclei and not with the electron cloud. Large samples can therefore be analysed without any need of sample preparation. This is crucial in the characterization of valuable archaeological and art objects, whose integrity must be warranted and preserved during the analysis (Rinaldi *et al.*, 2002).

As an example, we briefly discuss the interpretation of the manufacturing techniques of prehistoric copper axes, based on the measured metallographic textures measured by neutron diffraction (Artioli *et al.*, 2003). In the case of metals, texture studies by reflected light metallographic analysis have long been employed in the characterization of the metallurgical processes, both for recent, historical, and prehistoric artefacts (Scott, 1991). It is known that cast, cold-worked, and



Fig. 2 – Experimental pole figures compared to recalculated ones (left) and recalculated pole figures for principal crystallographic directions (right). Lineation N-S, foliation in the plane. Equal area projection.

hot-recrystallized metals show rather different and well recognizable texture patterns due to the preferred orientation of the crystallites induced by crystallization and local thermal or mechanical stresses, so that the metallographic texture analysis may help in the interpretation of the manufacturing process. However, the conventional metallographic methods of analysis by optical microscopy require polishing and etching of the metal surface to evidence discontinuities such as crystallite boundaries, structural features, and defects. The technique is therefore invasive and when the surface of the object is altered due to corrosion sampling requires penetration into the bulk of the metal by deep etching or even drill coring. It can not therefore be applied to unique finds such as the Iceman's axe (http://www.archaeologiemuseum.it/f06 ice u k.html) (Fig. 3).



Fig. 3 – The copper axe of the Iceman, found with the mummy in the Similaun Mountain Group. Dated to about 3200 BC, it is the only prehistoric copper axe found with the original wooden handle and ropes.

In the present study the interpretation of the observed crystallographic textures, besides the available metallurgical literature, relies on a series of laboratory copper standards prepared by us by different processes (casting, cold working in one and two directions, thermal annealing after cold working, final working at room temperature after the annealing cycle, etc.), which were carefully characterized both by metallographic and by crystallographic texture analysis.

The neutron data were collected at beamline D20 of ILL, Grenoble. Details of the measurements and of the data analysis are reported elsewhere (Artioli et al., 2003). The analysis of the neutron powder diffraction histograms and the polar figures resulting from the texture analysis performed on the central part of the body of the Iceman's axe shows very large copper crystallites and the total absence of texture (Fig. 4), indicating a nearly random orientation of the crystallites and production of the object by pure casting in a mold. The same lack of texture has been observed in the copper axe from Kollman, Bolzano, and in the modern replica of the Iceman axe produced by casting, which was kindly made available by Prof. G. Sperl, and which clearly shows under the microscope (Fig. 5) the common dendritic texture derived from rapid crystallization in the mold. The pole figures of both samples show no measurable deviation from a random distribution of the copper crystallites. Direct casting process with no mechanical hardening is the only reasonable interpretation of the observed features, as there is no simple way in which the textures induced by cold or hot working of the object could be destroyed by successive processes, such as for example extensive thermal annealing. Perusal of the metallurgical literature and our laboratory experiments clearly show that if slip systems are produced by volume reduction during mechanical shaping of the object, then they are the preferred sites of high temperature nucleation, and the copper crystallites arrange in well defined orientations forming the socalled «cube texture» (see Rollett and Wright



Fig. 4 – Pole figures resulting from the diffraction measurements of the central part of the copper axe of the Iceman. The figures are featureless, indicating the total absence of mechanical working or thermal annealing of the axe.



Fig. 5 – Copper dendritic crystallites observed by optical metallography on a polished section of the modern replica of the Iceman axe. The replica was produced by simple casting in a bivalve mold.

in: Kocks *et al.*, 1998). This is indeed the remarkably well developed texture observed in the Castelrotto axe (Fig. 6), which shows an extreme preferred orientation of the copper crystallites, resulting from extensive thermal annealing after substantial mechanical working of the object.

CONCLUDING REMARKS

The structural characterization of geological samples can not ignore the quantitative textural determination of the fabric and microfabric features involving the mineral phases, as these largely control the physical and mechanical

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Fig. 6 – Exceptionally well developed cube texture in the pole figures resulting from the diffraction measurements of the central part of the copper axe from Castelrotto. The texture is typical of extensive recrystallization due to thermal annealing after mechanical shaping of the object.

properties of the rocks. Texture information is essential to correctly interpret any tensor property, such as seismic velocities, compressional strength or electric conductivity, and to reconstruct the rock deformation mechanisms acting under different P,T regimes.

It is argued that the conventional methods of extracting experimental pole figures from a number of sample orientations, extensively applied by engineers in the case of metal samples of well known chemical composition and characterized by high sample and crystal symmetries, are often impossible to be applied to complex geological materials. Recently developed methods of combined Rietveldtexture analysis from diffraction data are based on the use of the full diffracted intensity profiles, instead of single peaks. They allow a more complete and flexible type of texture analysis, insofar the crystal structures may be refined simultaneously with the texture components, they may be applied to complex polyphasic samples exhibiting strong peak overlapping, and they may be combined to the residual stress analysis of some of the phases present in the sample.

It is also shown how modern texture analysis may be successfully applied to archaeometrical problems. Neutron diffraction texture analysis is to be considered a totally non-invasive stateof-the-art technique for the characterization of archaeological and art objects.

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