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Utilization of spatial resolved qualitative texture assessment method on an object of the Seuso Treasure

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Abstract. Centerless X ray diffractometers are specially designed for nondestructive residual stress measurements. The similarity between the X-ray diffraction based stress and texture measurement methods has led us to introduce measurement method for nondestructive (sample cutting-free) stress measuring devices, to describe the texture. After the determination of identical lattice plane series distribution with centerless and conventional diffractometers, advanced anisotropy measurements techniques were invented. The new examination methods were applied for a Late Roman silver artefact, the Perfume Box from the Seuso treasure. The spatial resolved qualitative assessment of the texture provided valuable insight on the processing of the material and manufacturing of the different parts of the artefact. The developed techniques are completely nondestructive, therefore the examined artefacts suffer no harm. The methods inherit the privileges of centerless diffractometers such as no object size limits.

1. Introduction

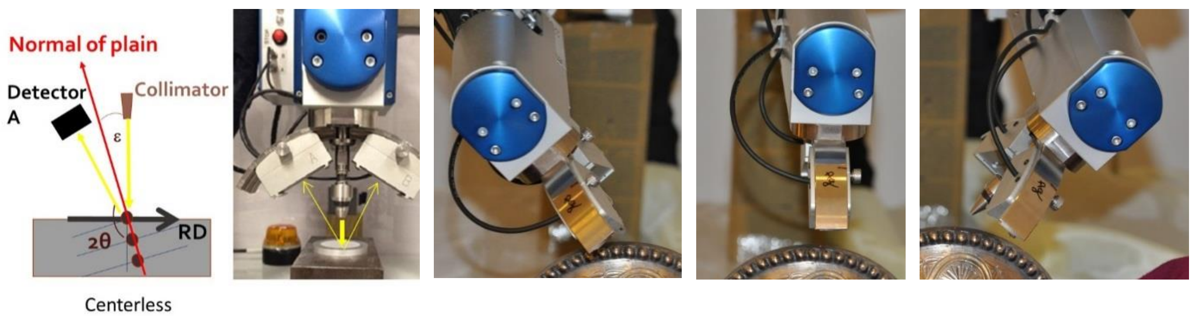
For archeological finds, especially high-value objects, only nondestructive testing modes are permitted as test options. This is true even though destructive testing methods give answers to many questions that cannot be achieved by nondestructive means [1]. For example, in the case of metal sampling, a simple light or an electron microscope examination can be used to give a clear evidence of the production and manufacturing technology based on microstructural characteristics [2]. Since the conditions of metal forming are clearly imprinted into the objects, it appears in the state of residual stress and texture [3,4]. Therefore, nondestructive mapping of these properties can provide important information. The two characteristics can be measured very well with X-ray diffraction technique. Classical powder diffractometers require Bragg-Brentano layout with a limited sample size and geometry satisfying the focusing conditions. It provides some degree of freedom when the diffractometer can function with a Göbel mirror and parallel beam operation [5]. Then, curved surfaces can be tested, centered on the pattern plane, which requires a careful and time-consuming measurement setup. The sample size is also limited here, but we get information about the entire diffraction angle range.

Centerless diffractometers [6] do not require sample cutting and virtually have no sample size limit. Such diffractometers are designed to determine residual stress in a nondestructive manner of machine parts, even in field conditions. The similar actions, tilting and rotating movements during residual stress and pole figure measurement led to the idea of developing the texture test method for centerless diffractometers. A single limitation of the device is that, for a given setting, a narrower diffraction angle range, namely, a higher Bragg angle range can be mapped in contrast with standard powder

diffractometers. The development of the test and evaluation methods are continuing to extend the features of the equipment and to extract information beyond the residual stress.
The Seuso treasure is one of the most famous silver collection from the Late Roman period. It is composed of 14 silver vessels and copper cauldron in which they were found. The name comes from the original owner, Seuso to whom the treasure is addressed in a couplet inscribed on the niello-inhalid Hunting Plate. The objects were probably manufactured in the Roman Empire at various points between A.D. 350 and 450. The Cascet- Perfume Box- having a cylindrical body a conical lid and an interior disk pierced with 7 holes for toilet flasks. The body and lid of the Cascet were both manufractured from single sheet of silver. No evidence of any joints can be detected. Ultrasonic measurements indicate that the Cascet is made of thin silver measuring only 0.5 to 1.5 mm in thickness. Because of the areas of high relief are not thinner than the background, suggesting extensive working of the silver from both outside and inside [7,8]. However, no other evidence about the deformation processes during shaping had been revealed so far. Because of the very high value of the objects, no destructive investigation could have been performed to reveal the microstructure. In this research work, the aim is to extract as much information as possible about the material, the production and the afterlife of the objects, from the large amount of diffraction data obtained during standard residual stress measurement carried out without destruction. In the present case, the focus is to determine the texture characteristics from the residual stress measurement data by visualizing the characteristic sections of pole figures.

2. Experimental

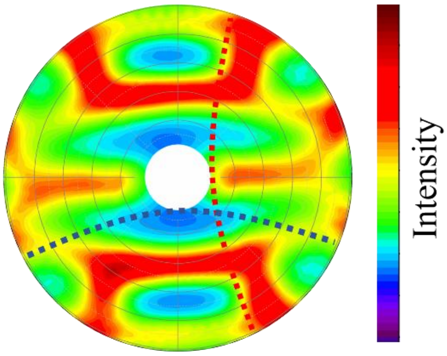
The standardized modified $\sin^2\psi$ residual stress measurement method (the beam path is realized according to Figure 1) was used to determine the interferential function in different tilting position. The measuring parameters of the Stresstech G3R centerless diffractometer were the followings: exposure time is 5 seconds; accelerating voltage 28 kV; current 8 mA; collimator 4 mm in diameter; detector arc 50 mm; X ray source Cr, tilting range -45 / + 45 °; number of tilting made 5/5. The intensity distribution of {222} and {311} reflections of silver were measured for the texture analysis. To interpret the intensity data of residual stress measurements in terms of texture, it must be transformed to be comparable with the standard representation method. The effect of tilting on the intensity value (different absorption due to the different path length) must be also corrected. A measurement with the same tilting was done on the silver powder and a correction function was calculated and applied on the stress measured data.



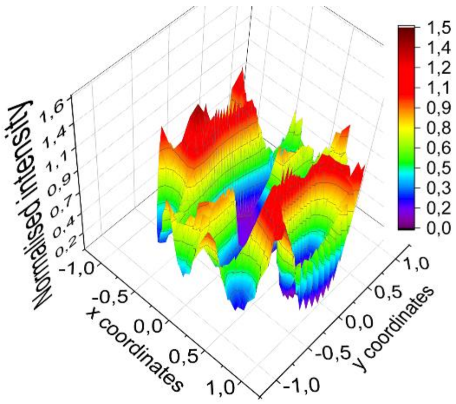
(a) (b) (c) (d)

Figure 1. The beam path and operating mode of the centerless diffractometer (a) schematic and photo in Ω mode, A is the position sensitive detector. Different ψ tilting positions in ψ mode (b) -45°, (c) 0°, (d)-45° during stress measurement.

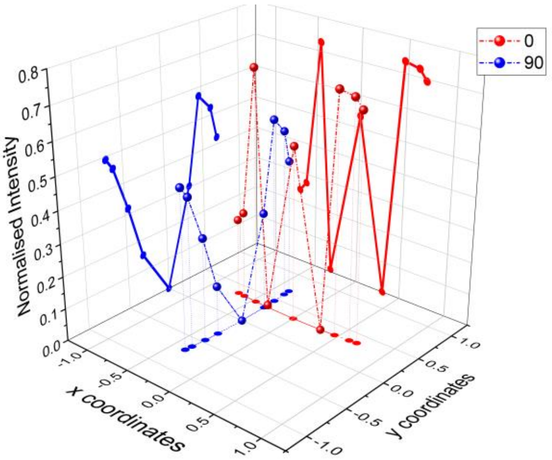
Furthermore, due to the performed standard residual stress data acquisition, only a few intensity data point is available from one measurement location, therefore, the standard χ-sections of the pole figure cannot be reconstructed. Another problem is that in the classical representation mode, the intensity distribution is indicated by colors or contour lines, which is not feasible if only a fraction of the measured pole figure is available. Therefore, it is necessary to leave the characterization with the standard polar coordinate system (χ,ϕ) and represent measured data, in the 3D Cartesian coordinate system.



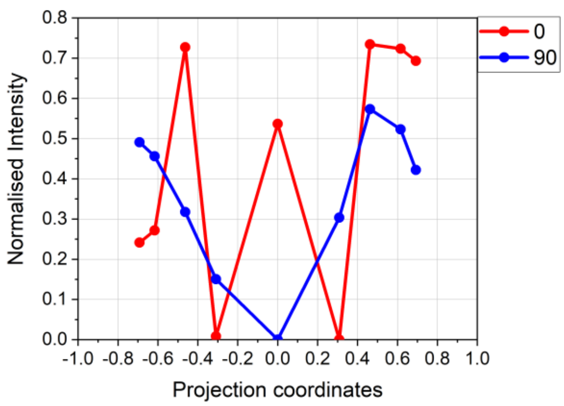
(a) {311} pole figure of rolled aluminum sheet, dashed lines represent data obtained from stress measurements



(b) The same {311} pole figure than (a) in Cartesian coordinate system



(c) Set of one colored data originated one stress measurement point on the {311} plane in the same Cartesian system than (b). 0 - in horizontal, 90 - in vertical direction



(d) Compare the projection curves of (c), representing the section of {311} pole figure in horizontal (0-red) and in the vertical (90-blue) direction

Figure 2. The procedure of visualization for qualitative texture description of a rolled aluminium sheet

The method of the calculation and visualization of the specific section of the pole figure is introduced through an example of a cold rolled aluminum sheet shown in Figure 2. After the determination of the identical lattice plane series distribution in centerless and in the conventional diffractometers, the {311} pole figure was measured with the conventional diffractometer Figure 2. a), while two data series (dashed line) of the {311} pole figure were obtained with the centerless diffractometer during stress measurements in modified ψ mode by tilting about the horizontal direction (Figure 2. a, dashed red line,0°) and vertical direction (90°, blue dashed line). The pole figure measured data was transformed from the polar coordinate system (χ,ϕ) to the 3D Cartesian coordinate system (x,y) using the (1); (2); (3) equations (Figure 2(b)):

$$x_i = \chi' \cos\phi_i \tag{1}$$
$$y_i = \chi' \sin\phi_i \tag{2}$$

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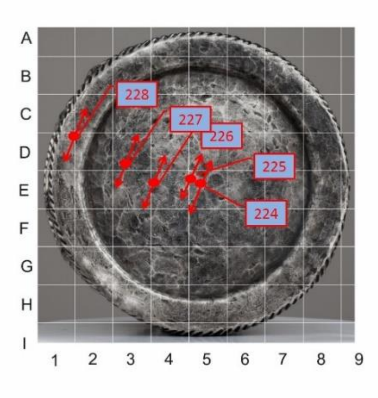
$$\chi'_{\cdot} = \frac{\chi_i}{\chi_{max}} \tag{3}$$

Due to this transformation, the value of χ is between -1 and +1. The value of z intensity coordinate was normalized within one measurement. The data obtained with the centerless X-ray diffractometer are visualized in the Cartesian system Figure 2(c) dashed lines. Because of the low information of the 3D visualization, the projection into the x-z and y-z plane were introduced (blue and red solid line curves, Figure 2. d). The two projection curves clearly represented the different texture features. If the measurement points of an object that were determined during stress measurement were measured in the same measuring direction related to the coordinate system of the object, the following analysis can be performed. Refer the selected measuring direction to a particular direction of the pole figure. Calculate and transform the measured stress point into the Cartesian coordinate system. Perform the correction for the intensity value based on the powder data was determined at the same tilting position. Visualize and compare the projection curves for each measurement point.

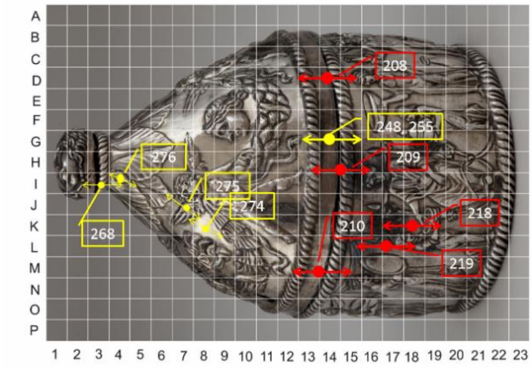
3. Results and discussion

Measurements have been carried out at many locations on the Perfume Box. In the gridded photo (Figure 3), the measurement locations and the direction are symbolically marked according to the tilting position, which is very important when evaluating the texture results. The Perfume Box consists of a cylindrical body (B) and a conical cover (C) (Figure 3(b)). Axial measured direction was applied on the body and the cover while tangential direction was used on the bottom of the body (Figure 3(a),(b)). Since the measured directions were identical at every measurement points, the calculated data are comparable.

The measurement points numbered from 205 to 275 can be divided into six groups depending on their locations on the artefact. From the machining point of view, (i) the bottom of the cylindrical body, (ii) the cylindrical body and the envelope of cone, (iii) the edge of the body and the cover, (iv) and the embossment pattern on the body and covers can be separated. The edges of the body and cover are overlapped on the gridded photos. The points 224-228 on the bottom of the box (B-i), the points 206-213 on the edge of the body (B-iii), the points 214-223 on the cylindrical body (B-ii), while the 270, 271, 272, 273, 274 points on the cone cover (C-ii), the 267,269,275 points on the cover embossment (C-iv) and the 247-255 points on the edge of the cover (C-iii) were measured. The position of the measurements 201 and 254 in the edge of body and the cover respectively are shown in Figure 3(c),(d). Figure 4 and 5 show the results of measurement points. The bottom plate data (Figure 4(a)) are distinctly different from the results obtained at the other measurement sites, both plain series show a non-characteristic, a quasi-isotropic structure. There are two reasons for this. One is that the deformation did not have a distinct orientation, which is a logical consequence if thinning was done with hammering. The other possibility is that heat treatment was applied after the deformation. Similarly, it has an uncoordinated texture of the data measured on the embossed areas of the cover (Figure 4(b)). This obviously comes from a non-preferred shaping direction which is also logic if any kind of shaping methods likes chasing, vibration embossing or die hammering were applied. The data on the body, however, have a clear deformation feature for each of the plane series in each measured point (Figure 4.(c)). Data from the plain series {222} shows larger sensitivity to the degree of formation on a more fragmented surface, such as the 214 measurement point, which is surrounded by embossed sections. In the case of the cover, this difference is even more pronounced (Figure 4(d)). The strongest forming characteristics are shown by both edge of body and the cover (Figure 5(a), (b)). The "earing" appearance is clearly visible as it can be seen in the measurement points 210 and 254 on the body and the cover respectively (Figure 3(c), (d)). The maximum normalized intensity values were also measured on the edge of the cover and the body. Since silver is a very well-formed metal, it is suitable for many shaping operations. The shaping technology of these archaic objects is radically different from today's industrial metal technology, so the practice in those industrial problems does not help in understanding the resulting texture image. However, it is clear from the results that the bottom is in an isotropic shaped or partially softened state. While in contrast, a texture-like pattern of unidirectional formation was visible on the body and cover, which increased even more strongly along the edges, that is likely to be the result of a metal spinning operation.



(a)Locations of measurement points on the bottom



(b)Typical measurement points on the body and cover

(c)Measuring the edge of the body

(d)Measuring the edge of the cover

Figure 3. The measurement points on the Perfume Box

4. Summary

A new texture characterisation method was developed based on the data of nondestructive (sample cutting-free) residual stress measurement performed with a centerless diffractometer. The method was used to describe the texture feature of a valuable Late Roman silver artefacts, the Perfume Box, the one part of the Seuso treasure. The intensity distribution of the silver {222} and {311} plain series were used for the texture analysis. Despite the few measuring data in one location which were determined during residual stress measurements it can be concluded that the method provided consistent and relevant texture information on the different part of the Perfume Box. Based on the evaluations it is possible to distinguish clearly the metallic state of the bottom, the side wall, the embossment and the edges of the box. The bottom plate and embossment data are distinctly different from the results obtained at the other measurement sites, both plain series show a non-characteristic, a quasi-isotropic structure. The body and the cover of the box have a clear deformation feature for each of the plane series. The strongest forming characteristics are shown by both edge of body and the cover, earing was also visible. These results were confirmed by several parallel measurements on the similar part of the Perfume Box. The results of these evaluations give guidance not only to the production technology, but also to the possible future target texture measurements. Future plans can also help in the technological reconstruction by determining the texture images that can be linked to the targeted shaping.

Acknowledgements

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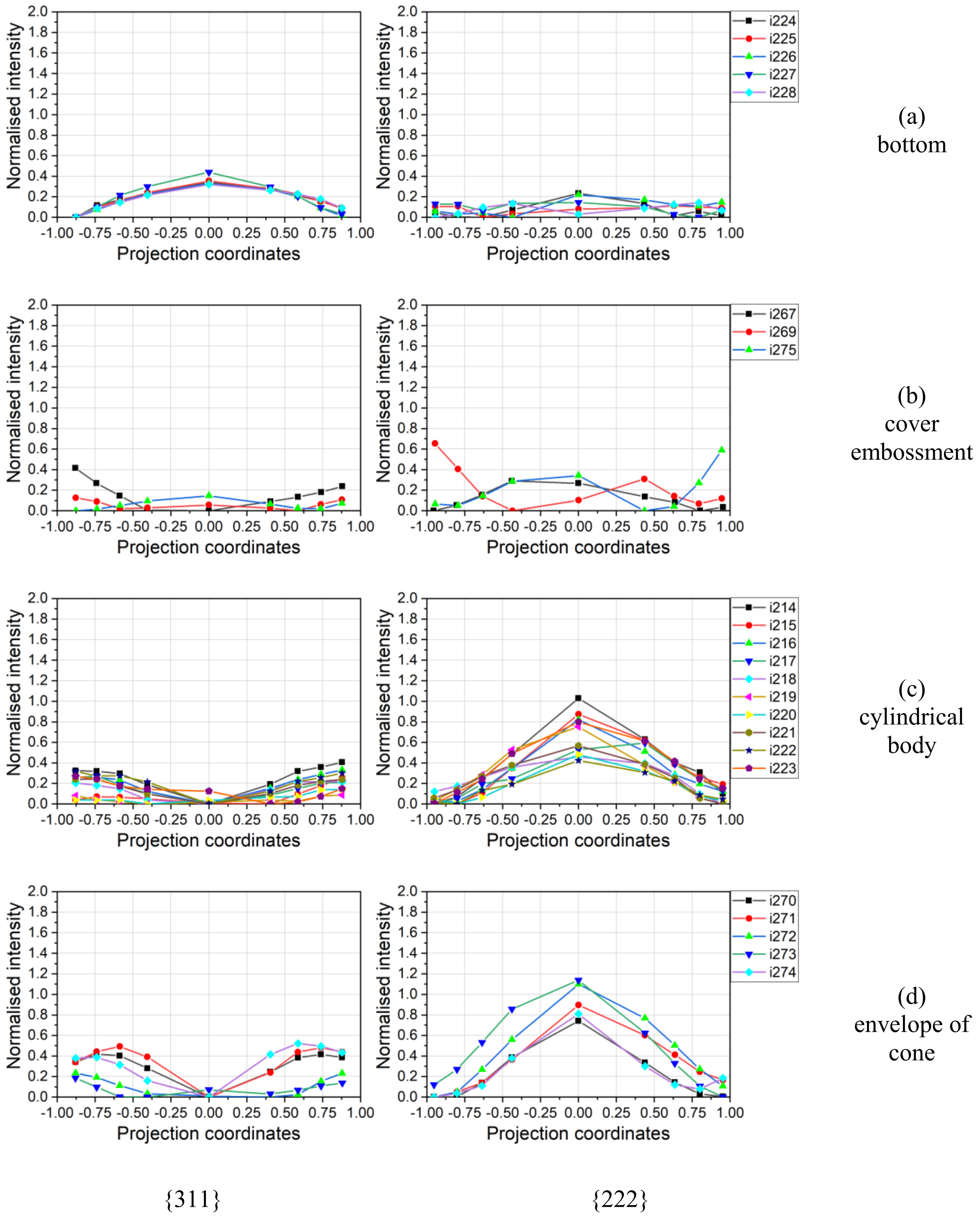
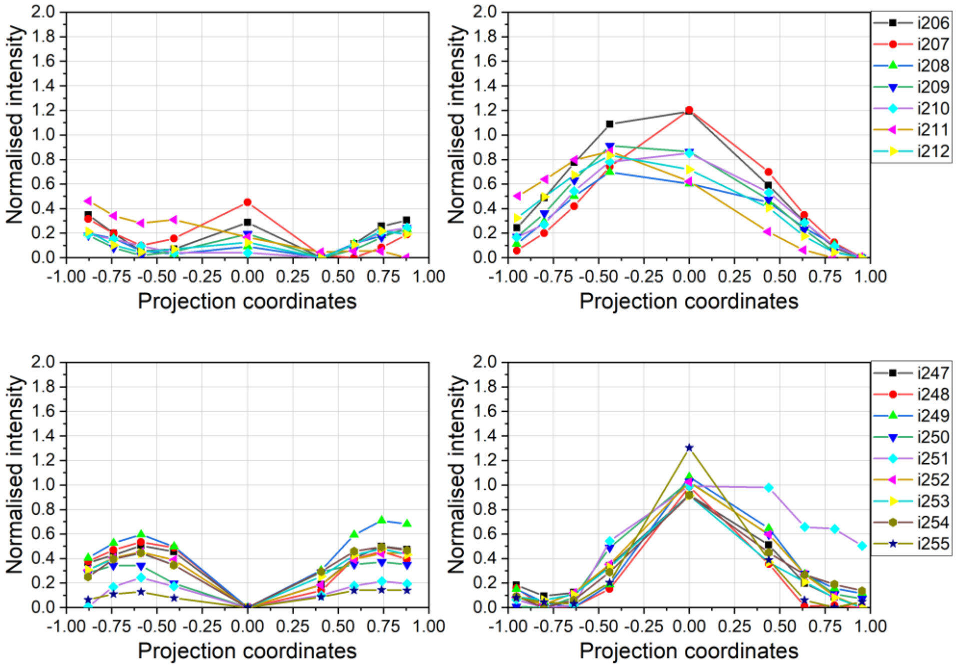


Figure 4. The calculated texture feature (projection curves) of {311} and {222} on the different part of the Perfume Box



(a) edge of the body

(b) edge of the cover

{311} {222}

Figure 5. The calculated texture feature of {311} and {222} on the different part of the Perfume Box

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