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Novel applications of centreless X-ray diffractometers for non-destructive pole figure measurements

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Abstract. Centreless X ray diffractometers are specially designed for non-destructive residual stress measurements. The similarity between the X-ray diffraction-based stress and texture measurement methods led us to introduce a new method on non-destructive (sample cutting-free) stress measurement devices to studies on crystallographic texture. The method that determines texture characteristics from residual stress data has recently been introduced. Afterwards, a method with which pole figures (fully equivalent to conventionally measured pole figures) at high Bragg-angles can be constructed with centreless diffractometers were developed. The paper gives an illustration of the new methods' utilization on the field of archaeometry. In that area, only non-destructive testing modes are permitted. In the case of archaeological finds, knowing the crystallographic texture assists to reconstruct the ancient process technology (hammering, rolling, annealing) that was applied during the manufacturing. The new methods are suitable to be used on valuable and unique objects both in the automotive industry, archaeometry and in the space applications.

1. Introduction

For archeological investigations, only non-destructive testing methods are permitted [1-2]. Neutron diffraction is a commonly used method for examining archeological objects, although this characterization method is not easy to access, and the sample is activated during the test. Neutron diffraction gives information about the whole, or a large volume of the object [3]. If local investigation is needed, the use of X-ray diffraction is a good solution [4]. If the goal is to obtain information from a very high resolution (from small volume), electron back scattered diffraction (EBSD) is a commonly used test method [5]. In the latter cases, however, a sample must be cut. A new, non-invasive texture characterization technique based on X-ray diffraction using centreless diffractometers have been developed [6]. Such diffractometers are designed especially for residual stress measurements which causes displacement in crystalline lattice and results in a shift of Bragg-angles. Centreless diffractometers are able to detect the shifts, the peak intensity and full width half maximum precisely. For accuracy, the operational range for centreless diffractometer is typically at high Bragg-angles (2θ). Since tilting and rotation are required for both texture and stress measurements, it led to the idea of developing the texture test method for centreless diffractometers [7-8].

In metals, crystallographic textures develop after plastic deformation and heat treatments. One of the most common ways to characterize texture is to measure pole figures. Pole figures indicate the angular distribution of given crystallographic planes on a reference coordinate system.

In the industrial practice it is common to optimize the technological parameters based crystallographic texture for better material properties.. In archeometry, the interpretation of texture is suitable to determine and reconstruct the technological processes of question [2,5,9,10].

The Seuso treasure is one of the most famous silver collection from the Late Roman period. The body and lid of the Perfume Box were both manufactured from a single sheet of silver. No evidence of any joints has been detected. Ultrasonic measurements indicate that the Perfume Box is made of thin silver sheets with only 0.5 to 1.5 mm in thickness. Since the areas of high relief are not thinner than the background, an extensive working of from both outside and inside is supposed [11,12]. However, no other evidence about the deformation processes during shaping had been revealed so far. Because of the very high value of the objects, non-destructive investigation is the only acceptable method to reveal the microstructure. In an earlier work of the authors [13] the residual stress dataset was used to obtain information about the texture. There are many examples in the literature that not the full pole figure, but only specific components are necessarily needed for adequate information about the texture [14-16]. In this paper, data from specific directions are compared to pole figures from the Perfume Box of the Seuso treasure.

2. Experimental procedures

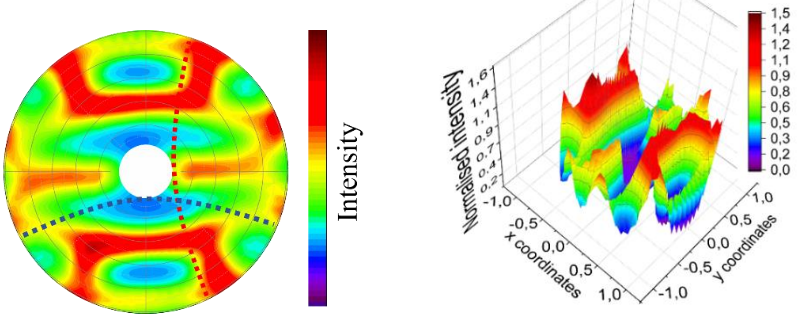
The standardized $\sin^2\psi$ residual stress measurement method was used to determine the interferential function in different tilting positions. The measuring parameters for the applied Stresstech G3R centreless diffractometer are as the following: exposure time is 5 s; accelerating voltage 28 kV; current 8 mA; collimator 4 mm Ø; detector arc 50 mm in modified ψ mode; Cr source, tilting range -45 / + 45 °; number of tilting: 5/5. The {222} and {311} reflections of silver were measured for qualitative 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 was corrected with silver powder. Due to the standard residual stress data acquisition, only a few intensity data points are available, therefore, the χ-sections cannot be reconstructed.

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 (figure. 1). 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 [13]. Refer the selected measuring direction to a particular direction of the pole figure, for example the rolling direction. Calculate and transform the measured point into the Cartesian coordinate system. Perform the intensity correction. Visualize and compare the projection curves for each measurement point. The signal of the two detectors were identical, therefore only the projection curve of detector A is shown.

By extending the stress measurement method, it is also possible to determine pole figures. This is possible in a variety of ways. One is to determine the measurement directions of the centreless diffractometer in accordance with the points of the conventional pole figure, which is the so-called modified X (CHI) method [6]. The other option is when the pole figure coordinates of the applied stress measurement positions (tilting and rotations) are determined, called the reverse modified X (CHI) method. This mode is described in detail in another manuscript of this journal [17]. Since the beam path differs from centreless and conventional diffractometers, the coordinates (tilting and rotation angles) used in the tests are not the same of the two measurement methods, so the equivalent positions for both modified and reversed modified cases had to be determined.

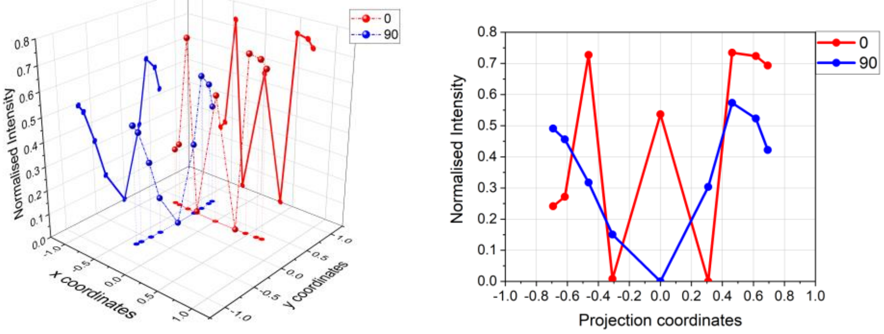
3. Results and discussion

The Perfume Box consists of a cylindrical body and a conical cover. The edges of the body and cover are overlapped on the gridded photos (figure. 2) where the residual stress measurement locations and the direction are marked according to the tilting positions. Measurement points 206-212, 214-223 and 224-228 are from the edge, side and the bottom, respectively. Axial measured direction was applied on the body (edge and side) while tangential direction was used on the bottom of the body. Since the measured directions were identical at every measurement point on the same part, the calculated data are comparable.



(a) {311} pole figure of rolled aluminum sheet,

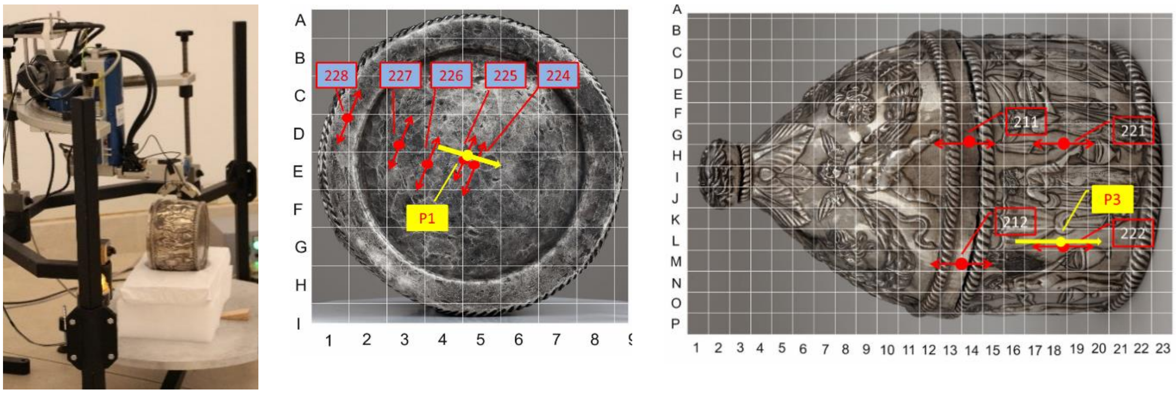
(b) The same pole figure in Cartesian coordinate system



(c) Solid lines are the projection curves of the dashed lines in the Cartesian system

(d) Comparison of the projection curves

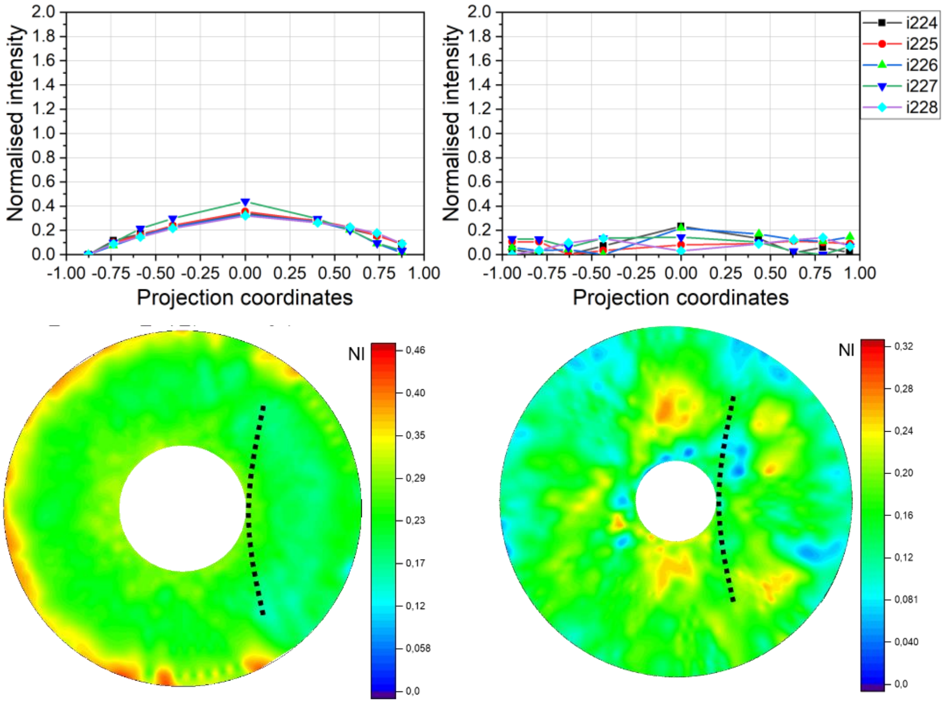
Figure 1. Procedure for qualitative texture description [13]. Dashed lines represent data obtained from stress measurements.



(a) (b) (c)

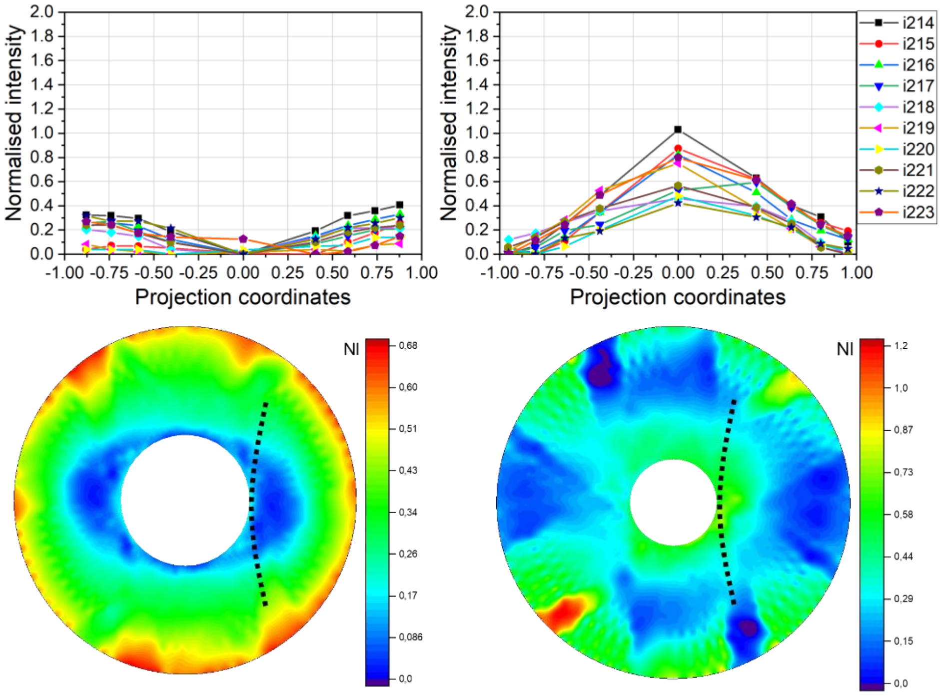
Figure 2. Investigation of the Perfume Box**.** (a) Onsite texture measurement with centreless diffractometer. Measurement points and tilting directions (b) on the bottom (224-228 stress, P1 pole figure) (c) on the side (221,222 stress, P3 pole figure) and on the edge (211,212 stress) of the body.

Pole figure measurements have been also carried out on the bottom (point P1) on the edge (P5) and on the side (P3) of the body. The starting point of the pole figure (conventionally called rolling direction) is also indicated on the gridded photos. The P3 and the 222 measurement point give data from the absolutely same location of the side of the body. While P1 and 225 are identical on the bottom.



{311} {222}

Figure 3. Projections curves and pole figures of the bottom part of Parfume Box



{311} {222}

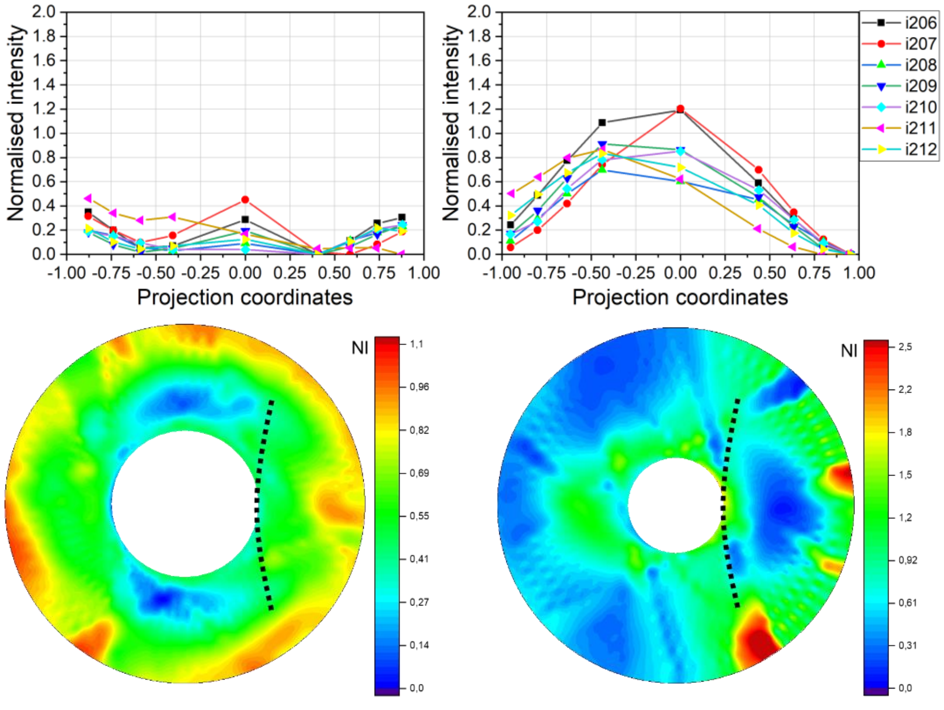
Figure 4. Projections curves and pole figures of the side part of Parfume Box

figure 3-5 show projection curves and pole figures from different parts of the object. The measurement points of the projection curves are marked on the pole figures by the dotted curves. The bottom plate data are distinctly different from the results from other parts. Both plane series show a quasi-isotropic structure (figure. 3). The results of pole figures show similar results. The non-textured nature is in line with the assumed final hammering (patenting) of the bottom part. 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.

Data from the side, however, show a clear deformation feature (figure. 4). Compared to the bottom data, the pole figures are much more characteristic. The direction of generatrix is the direction of the poles for 12 hours. The pole figure does not resemble the known forming operations. It can be clearly stated that the deformation of the side differs from the shaping operation of the bottom and that the side is certainly not shaped by hammering.

The strongest forming characteristics are found at the edge of the body (figure. 5). Comparing to the pole figures on the side, the polarity of the figures is the same for the same index but a 90° rotation can be observed. It is evident from the fact that the starting position during the measurement was also rotated by 90°.

The shaping technology of these objects is radically different from today's industrial technologies, so the practice in those industrial problems does not help in understanding the resulting texture. However, it is clear from the results that the bottom is in isotropic, thus mildly shaped or partially softened state. While in contrast, a texture-like pattern of unidirectional formation was visible on the side, which increased even more strongly along the edges, that is likely to be the result of a metal spinning operation.



{311} {222}

Figure 5. Projections curves and pole figures of the edge part of Parfume Box

4. Summary

A fast texture characterisation method by projection curves was developed based on the data of non-invasive residual stress measurement performed with a centreless diffractometer. The method was used to describe the texture feature of the Perfume Box, the one part of the Seuso treasure. It can be concluded that the method provided consistent and relevant texture information on the different part of the Perfume Box. It is possible to distinguish clearly the metallic state of the bottom, the side and the edges of the box. The bottom plate data are distinctly different from the results obtained at the other measurement sites, both plane series show a non-characteristic, a quasi-isotropic structure. The side of 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. The results of these evaluations give guidance to the production technology.

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