### ON MICRO-SCALE STRAIN MEASUREMENTS USING TIME-BASE PHOTOGRAMMETRY

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# 1. Introduction

From the time of Boyde's /l/ practical application of scanning electron microphotogrammetry for the studies of developing tooth enamel surfaces, numerous further applications were elaborated. A review of papers developing the method of microphotogrammetry was made by Boyde /2/. Some of them were published in the Proceedings of foregoing Congresses of the International Society for Photogrammetry, Commission V, /3/, /4/, /5/. Very interesting are the applications of microphotogrammetry to materials science. Krasowski /6/, /7/ applied microphotogrammetry to the morphological characterization of fatigue crack surfaces. At present similar procedure forms part of the LINK Analytical System AN 10000, the so-called Stereopackage /8/.

Another important application of photogrammetry in this field is the analysis of displacements and strains in deformed structures, both macroscopic and microscopic. These applications are closely related to the time-base method, in which two photographs are taken under different loading states, the first before and the second after loading, by the same camera. Such application has been known since 1913 /9/ and some other cases and bibliography on this subject are described in the paper by the author /15/. Similar conditions for taking a couple of photographs are provided in a microscope. If provided with an adapter to carry out the deformation of a specimen inside the microscope, it is possible to take photographs of the material structure before and after deformation. Two applications in this field are known. The first was elaborated by Davidson /11/, /12/, the second by authors /13/.

This paper gives essential data about the geometry of representation in SEM and its relation to the time-base method, improvements and further applications in this area.

2. The Time-Base Method /TBM/ and Evaluation of Microscopic Photographs.

The TBM is often used for the determination of small changes in the position or the dimensions of objects. It is called also the time base photogrammetry /14/, /10/. Although the photogrammetric analysis of a couple of photographs is carried out in the same way as in stereophotogrammetry, the conditions for taking of photographs are different. The photographs must be taken in this case with identical external and internal orientation of the object and the camera.

The conditions of representation of microscopic objects are simplified, if the objects are planar and particularly if the

stability of the magnification ratio in a time are better in optical microscopes, because of their stiff optics. The electromagnetic lenses of the SEM afford worse conditions for the stability of magnification, as they are very sensitive to temperature and the electromagnetic changes of environment. Therefore, the calibration of microscopes is necessary.

Instead of errors of magnification a certain distorsion of the image occurs in the SEM, which is shown in Fig. 2. Provided this distortion does not change in time and the photographs made by the time-base method differ from each other only in deformation, then the ratio of the lengths of any line from the photographs taken before and after deformation is constant with the exception of second order quantities - in any place of the photograph. This is the basis of the possibility of using the SEM photographs for photogrammetric analysis of deformations, which are independent of the geometry of the SEM representation. The change of the image, similarly as the object itself, has two components: central and angular. The central component manifest itself also as strain; however, its distribution about the area of the photograph is not homogeneous. The angular component is antisymmetric with regard to the central component and does not influence the mean strain value.

The error due to the change of working distance is already included in the error due to the change of magnification. Small rotations of the sample, whether in respect of the optical axes, are centrally antisymmetric and do not influence the mean strain value determined from the photographs. They manifest themselves in the mutual rotation of both photographs, but with regard to the fact that they are incorporated in both the absolute rotation of the internal region - grain - and in the absolute rotation of its environs, they do not influence the relative rotation of the grain with regard to its heighbours, since they are mutually subtracted in the calculation.

3. Photogrammetric Analysis of Microphotographs and Preparation of Specimens.

The determination of paralaxes from a couple of microphotographs is the main task of photogrammetric analysis. A significant element of the method, on which the high accuracy of evaluation of paralaxes depends /the analysis is carried out in a type Stecometer ZEISS Stereocomparator with internal accuracy of 2  $\mu$ m, is the perfect signalization of measured points. In these conditions the theoretical accuracy of evaluation of mean strain from photographs sized 50/60 mm, is expressed by the value of

 $e = \frac{2 \times 0,002}{50} = 0,8 \ 10^{-4}$ , which can be considered as actual conditions for the coincidence of marks in a stereocomparator are worse than the theoretical ones, the real mean error of strain is greater.

Natural structural defects on the surface of some specimens, if they are sharp and bright enough, can be used for the analysis. The image in Fig.3, is the surface of a specimen of

process concerns the change of their dimensions only. In either case the photographs can be taken, using an optical microscope. The scheme in Fig. 1 shows the difference in projection, when the photographs are taken by a camera a/ and in a microscope b/. If a SEM is used and if the object of investigation is the same as before, i.e. relative changes of microobjects, it is possible - provided the representation properties of the microscope do not change within the real time interval concerned - to determine the relative changes of the microobject, regardless of the manner of representation, as shown in Fig.2.

Also magnification and position of the photographed microobject must be preserved.

With regard to the forces in the adapter, producing tensile deformation of specimens in the vacuum chamber of the SEM, small changes in the focussing of the microscope and in the position of the sample occur. In every loading step, i.e. when taking the photographs preceding and following deformation, the microscope must be refocussed. which results also in a certain small deviation in magnification, which is of the same sign and magnitude at the whole region of the photograph. This error is most significant, as it manifests itself as strain and influences the calculated value of the strain. A deviation in the photograph magnification, of pre- and post-deformation photographs of 1%, gives the same error in the measured strains, which is a very gross value for strain measurements. The SEM provides better conditions for high accuracy rectification of the mag-





Fig.2

nification ratio, after refocussing, to initial level, then the optical microscope. It is due to the greater working distance, i.e. the distance between the plane of the specimen and that of the lens, in the SEM. On the hand, the conditions for the pure polycrystalline Al, which was used for strain analysis described in /13/.

To get better results in respect of the accuracy of measurements of paralaxes from microphotographs, it is necessary to provide the surface of the specimens with artificial marks, gratings or points. The electron litography is a convenient technique to use for this purpose. The high quality of the image of these marks on photographs depends then on the characteristics of their litographically made originals, as well as on the conditi-



Fig. 3

ons of their photographing in a scanning electron microscope. The width of lines of the grating or the diameter of the points must be optimized with regard to magnification, contrast and brightness.

4. Evaluation of Strains from the Elements of the Photogrammetric Analysis.

With regard to imperfect signalization of the points on the microsurface of the specimen it was not possible to determine with sufficient accuracy the history of the displacement between the individual points and to construct the strain map. From geodetic methods, which could be used for the calculation of strains from the ascertained coordinates, the affine transformation has appeared most suitable. Its application makes it possible to determine the mean strain in an area covered by a set of points, see Fig.4. The affine transformation of plane

coordinates  $x_k$  into plane coordinates  $x_k$  is described by the system of linear equations

$$x_{i} = A_{ik} x_{k}^{\prime}$$
,  $i, k = 1, 2$ 

in which  $A_{ik}$  is a matrix of its coefficients, which are asymmetrical  $A_{ik} \neq A_{ki}$  and are identical with the gradients of deformation. The calculation of the coefficients was carried out by means of an algorithm in the form of a computer programme /16/.



Fig.4

In accordance with the basic relations of the theory of deformations subsequently the formulas for the tensor of finite strains  $e_{ij}$  and the tensor of finite rotations  $\omega_{ij}$  are obtained /13/

 $\mathbf{e}_{ij} = \frac{1}{2} \left( \mathbf{A}_{ki} \times \mathbf{A}_{kj} - \delta_{ij} \right), \quad \boldsymbol{\omega}_{ij} = \mathbf{A}_{ij} - \mathbf{e}_{ij} - \delta_{ij},$ 

where i,j,k = 1,2 and  $\circ_{ij}$  is the Kronecker symbol, with the value of 1 for i=j and 0 for i≠j. A practical realization of the process of evaluation of microdeformations from the pair of photographs consists of visual stereocomparator measurements and automatic processing of the measured data as far as the calculation of the coefficients of affine transformation and the components of the strain tensor and rotation tensor and their graphical representation.

5. Applications of Micro-Scale TBM in Strain Analysis.

The method described above, which was elaborated for strain analysis in material structures, was used in three applications so far.

In the case according to Fig. 3 the kinematics and deformation of a polycrystalline structure was investigated. The specimen used was prepared from the experimental material, aluminium of 99,85% purity. For the purpose of microscopic analysis a specimen of dimensions of  $0,5 \times 7 \times 30$  mm was made of this material. The photographs were taken on a type JSM 50A Jeol SEM and the deformation of the specimen in the vacuum chamber of the microscope was produced by a fixture with a bolt-controlled jaw movement. Neither the magnitude of overall induced deformation nor the force were measured directly. This circum-stance considerably limited the possibility of directly determining the character of the deformation process and the micro-scale deformations could be observed only in accordance with the increase and decrease of load. The diagram in Fig. 5a shows the loading process and the stages in which the microscopic photographs of the area sized 50 x 60 um were taken with the magnification of 1000. In accordance with the conditions which appeared in the stereocomparator as most suitable for the coincidence of the photographs taken in the SEM, the parallaxes were consecutively measured in 100 points. Their distribution about the picture is shown in Fig.l. The computational processing of measured data according to paragraph 4, yields for the quantities of  $e_{ij}$  and ij the values from Table 1, which mean the mean strains of the whole area of the photograph and the relative kinematic rotation of the grain from Fig. 3 with regard to its environs.

The history of the components of  ${\rm e_{22}}$  and  $\omega_{12}$  during the loading is shown in Fig. 5b.

Further application of micro-scale photogrammetry, without artificial marking of measured points, was the analysis of strain distribution around the notch in a tensioned strip /17/ shown in Fig. 6a.

TABLE 1 Values of strain tensor and rotation vector components Deformation Loading  $e_{22}10^2$   $e_{12}10^2$   $e_{21}10^2$ 3.128 75 -1.380 26 -1.380 26 Stage Interval  $\Delta_{e_{22}} 10^2$ e<sub>1]</sub>10<sup>2</sup> 0.131 06 -0.174 14 1 0102 1.844 60 -1.411 44 -1.411 44 0.332 46 0.000 00 2 0203 0.541 00 -3.321 30 1.978 83 -1.902 66 -1.902 66 3 0304 2.697 63 -0.833 88 0.032 77 0.032 77 2.868 88 -0.012 08 -1.956 47 -1.956 47 4 0405 0506 5 6 Rotation Loading  $\Delta \omega_{12} \omega^2$  $\omega_{12} 10^2$  $\omega_{21} \omega^{2}$ Stage Interval -1.232 58 0.181 51 1.232 58 1 0102 -0.179 65 -1.977 02 1.571 82 2 0203 -1.977 02 0.233 13 1.977 02 3 0304 0.132 38 -0.132 38 4 0405 -0.022 40 5 0506 0.019 50 6 10 ∆e<sub>22</sub>10<sup>2</sup> e 22<sup>10<sup>2</sup></sup> 3 2 Loading stage ۵ ω<sub>12</sub>10<sup>2</sup> 1 Δω<sub>12</sub>10<sup>2</sup> 2

### Fig. 5a, b

0

The specimens were made from AlFe composition; the diagram in Fig. 6c shows the notch opening vs. load. The photographs were taken by an optical microscope, of the mark of Reichert, with the magnification of 312,5, Fig. 6b. The evaluation of displacements, strains and rotations in the area sized 0.26x0.36 mm near the notch head, was processed acording to paragraph 4. Fig. 6d shows the field of the rotation vector in Specimen 1 after loading - point A in the diagram in Fig. 6c.

2

3

45 Loading stage



The accuracy of strain evaluation by means of micro-scale photogrammetry was tested by a comparison of two photographs of a non-deformed specimen, but with repeated refocussing of the microscope. The strain determined from these photographs represents the mean error for the assumed case of measurement and is given by the value of  $e = 3 \times 10^{-4}$ .

The third application under investigation is connected with the analysis of the stability of magnification in the SEM as it was explained in the first paragraph. The stability of magnification in the SEM is examined for some operational regimes - during a certain time interval, which we need for the experimental handling of the specimen in the vacuum chamber, for refocussing and for the changes of working distance, voltage and current. The examination was carried out again by means of repeated photographing of, now, perfect grating, applied by means of electron litography to the surface of an Si plate. Three kinds of these gratings are shown in the photographs in Fig. 7. Their analysis was carried out by means of affine transformation again.



Fig. 7

The initial coordinates are represented by the photograph taken in the time point "zero" and the others, runing coordinates are represented by the photograph of non-changed grating, taken after a certain time interval - 10 minutes. The paralaxes measured in these photographs represent the input data for affine transformation again. Its results give mean errors of magnification of the SEM for regimes pointed above. Some results of the analysis of the 10 minutes stability of SEM magnification are presented in Table 2.

Table 2. The 10 minutes magnification stability /18/ Grating Fig. 7 a) b) c)

cop	Jeol 733 - Superprobe		÷	÷ 2.10 <sup>-4</sup>		
r00	Tesla BS 340	÷3.10 <sup>4</sup>	÷	2,5.104	÷	1.10-4
lic	Philips		÷	2,0.10 <sup>-4</sup>		

We are deducing from these results, that the high accuracy of micro-photogrammetric measurements strongly depends on a rate of a line thickness to line spaces and on a good quality of microphotographs, i.e. a good quality of the grating so good image in the microscope-contrast and sharpness. With regard to the fact that it is possible to improve the technical conditions for the taking of photographs and for the coincidence of the points in the stereocomparator, it is also possible to approach closely the theoretical accuracy of these measurements. On the basis of these achievements it is possible to conclude that the time base method of photogrammetry represents a suitable means also for the analysis of deformations in a material microstructures.

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