Analysis of microdeformations in a structure of polycrystals

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The resulting mechanical properties of structural materials are influenced by their structure, i.e. by the interaction of microscopic particles which manifests itself by the origin of microstrain and microstress fields. Their study is the source of understanding of microdeformation mechanisms influencing the history of all deformation and failure processes. The present work is concerned with the determination of strain and kinematic rotations of grains in a polycrystalline structure. The analysis of microscopic photographs is carried out by means of the photogrammetric time base method. Its data are then processed by means of affine transformation; from the coefficients thus ascertained the components of strain tensor and grain rotation vector are determined.

1. Introduction

1.1. Structural aspects in the mechanics of materials

Mechanical properties of materials are influenced by their structure through the microdeformation mechanism which manifests itself by the non-homogeneity of microstresses and microdeformations fields. It is in the interest of the study of the laws governing the formation of mechanical properties of materials to analyse these fields. On the basis of the works by Rovinskij [1] and Dawson [2], in which small kinematic rotations of grains of a polycrystal under elastic deformation were proved roentgenographically and microscopically, the author [3] designed a mechanical model of a polycrystal with such properties (Fig. 1). This study is intended, on the one hand, to verify previous works and, on the other hand, to obtain approxiamte quantitative strain and rotation values of polycrystalline grains.

1.2. Problem and method of solution

For the assessment of the microscopic photographs of a polycrystalline grain taken before and after its deformation, the photogrammetic method was choosen. Its use for the anlysis of microscopic photographs was suggested in 1973 by Boyd [4]. However, its applications to the measurements of microscopic objects is connected with certain difficulties, due to the representation abilities of scanning electron microscopes (SEM). As far as the authors, know, this method has not been used for the analysis of microdeformations, although the aforementioned difficulties are partly eliminated. Some measurement errors can be also eliminated by a suitable processing of the results of photogrammetric analysis.

2. Photogrammetric analysis

2.1. Principle of stereophotogrammetry (SFGM)

The original and most widely used methods of photogrammetry is stereophotogrammetry, which uses the natural ability of the pair of human eyes to see three-dimensionally in the process of the so-called artificial stereoscopic perception. The artificial stereoscopic sensation is created in the observer's sight centre similar to the actual

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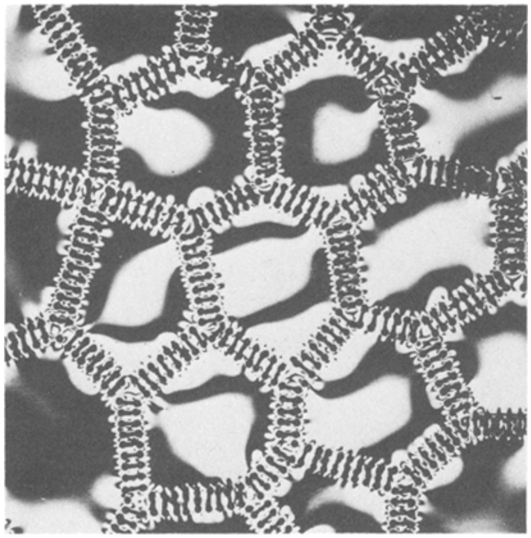

Figure 1 Model of an ideal polycrystal.

sensation. The difference consists in the fact that the analyser's eyes observe in the stereocomparator two photographs of the actual object, taken from the extreme points of the SFGM base, instead of the actual object.

2.1.1. *Topographic SFGM [5]*
In topographic SFGM the pair of photographs is taken by means of a base consisting of two rigidly joined survey cameras with perfectly parallel optical axes. Alternately the photographs are taken by means of two separate photo-theodollites which must be mutually oriented. On the whole the basis must be spatially oriented; apart from the elements of external orientation the elements of internal orientation also must be known and ensured with high accuracy, namely, the optical constants of the cameras and their image-forming properties. The photographs are taken either from land-mounted or by aerial means. The condition of accurate analysis of the photographs in the stereo-comparator is the perfect ensurement of the coincidence of the corresponding points in both photos by the analyser. To ensure this, conspicious and geometrically suitable marks must be made on the measured objects.

2.1.2. *Microscopic SFGM*
The great depth of focus of the SEM optical system enables one to take a couple of sufficiently sharp and contrasting photogrammetric pictures, which aroused the original interest in the use of SFGM in microscopy [6]. However, the photographs of the micro-object must be taken in two, perfectly accurately defined positions and with constant focusing of the microscope, which can be effected either by the displacement or the tilting of the sample. This endeavour, however, met with obstacles due to the representation abilities of the SEM. In contradistinction from optical microscopes, which do not distort the image of the object, but are not suitable for the SFGM pictures because of the small depth of focus, the optical system of the SEM results in the central and angular distortion of the picture, which can be neglected in the reconstruction of the picture of the micro-object only in optimum enlargement conditions, depending on the type of apparatus. For these reasons the application of SFGM in microscopy has not been very extensive so far [7, 8].

2.2. Time base method in photogrammetry
2.2.1. *Engineering photogrammetry*
The basic pattern of photogrammetric method is used also in other fields. Considerably frequent is the use of photogrammetry [9, 10] for the determination of small changes of position or dimensions of objects, which is called the time base method. Although the analysis of the photographs is carried out by the same method as in the case of SFGM, the conditions for the taking of the pictures are different. Both photographs must be taken from the same point in a certain time or loading interval and with identical external and internal orientation of the object and the carmera. However, any minute changes of position of either of them manifest themselves as measurement errors and are described later on.

2.2.2. *Microphotogrammetry with a time base*
The conditions of representation of microscopic objects are simplified, if they are two-dimentional, and particularly if the process concerns the change of their dimensions. In either case the photogrammetrric pictures can be made, using optical microscope. If a SEM is used and if the problem of investigation are not the actual dimensions and shape of the object, but the determination of its relative changes, it is possible – provided the representation properties of the microscope do not change with in the real time interval concerned – to determine the relative changes of the micro-

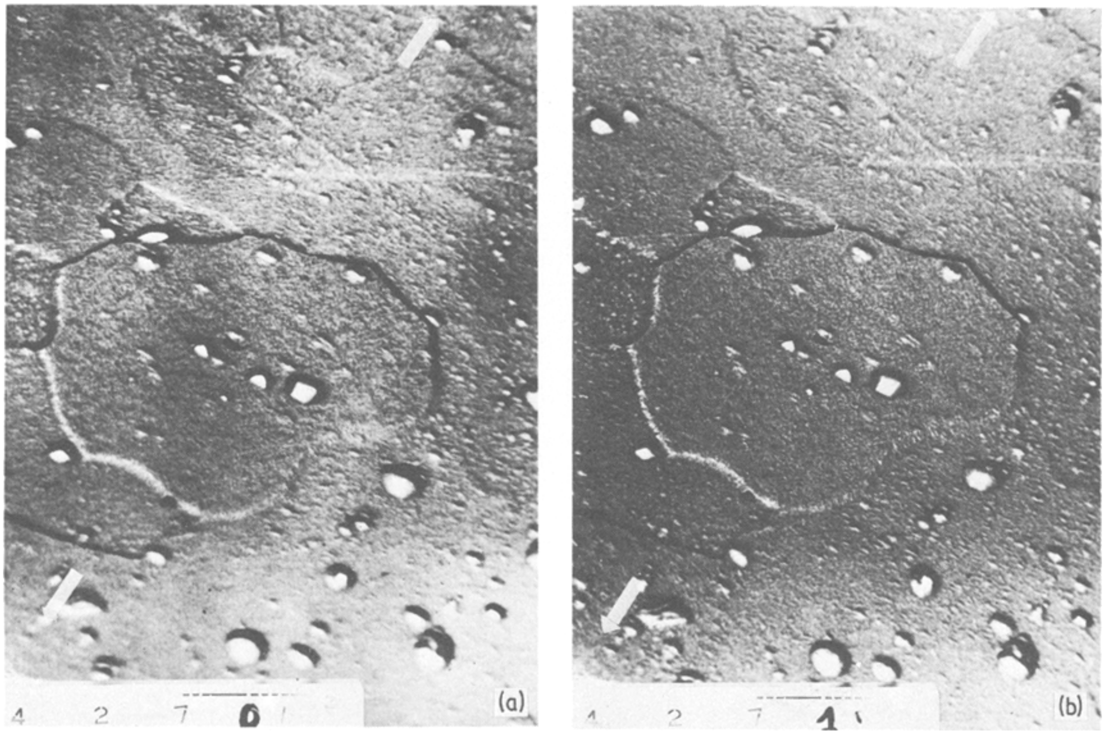

Figure 2 Photogrammetric pair of microscopic photographs.

object regardless of the manner of representation. On this basis, also, the analysis of the deformations of the polycrystalline grain has been effected, which is reported in this study.

2.3. Evaluation of microscopic photographs

2.3.1. Object of microstrain analysis

The purpose of the study is to determine the deformation of a polycrystalline structure on the level of grains, in the microscopic region sized $50 \times 60\,\mu$m, represented on a pair of microscopic photographs (Fig. 2) taken in a SEM with magnification of 1000×. The purpose of the analysis was to verify, on the central grain, the conclusions of preceding works [1, 2] pointing out mutual small kinematic rotations of grains of a polycrystalline structure in the course of elastic deformation.

2.3.2. Preparation of photographs and stereocomparator measurements

A significant element of the method, on which the high accuracy of evaluation of the photographs depends (the analysis was carried out in a type Stecometer Zeiss stereocomparator with internal accuracy of 0.002 mm) is the perfect signalization of measured points. In these conditions the theoretical accuracy of evaluation of mean deformations from the photographs of given dimensions is expressed by the value of

$$\Delta e = \frac{2 \times 0.002}{50} = 0.8 \times 10^{-4}$$

which satisfies the requirements. However, artificial marks thus defined could not have been made on the surface of the sample, and coincidence was based on natural defects of surface structure visible in the photographs, which resulted in a reduction of measurement accuracy. Some improvement could be achieved by an increase of the contrast of the photographs, which were taken on film and were not suitable for use in a stereocomparator. The photographs were therefore contact copied on hard photographic plates affording good contrast. After insertion of the photographs into the stereocomparator and their mutual orientation the coordinate x' and the parallax p_y of the photographs taken at the left end of the time or loading interval and the coordinate y'' and the parallax p_x of the photograph taken in the right end of the interval are measured. The remaining coordinates are then calculated from the equations

$$y' = p_y + y'' \quad \text{and} \quad x'' = x' - p_x.$$

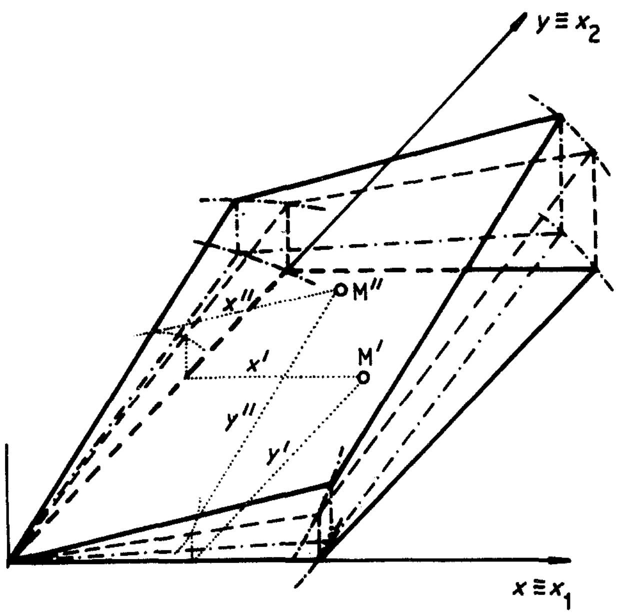

Figure 3 Schematic representation of affine transformation.

2.3.3. Evaluation of deformations from the elements of the FGM analysis

With regard to imperfect signallization of the points on the surface of the sample it was not possible to determine with sufficient accuracy the history of the displacement between the individual points and construction of the strain map. From geodetic methods which could be used for the calculation of deformation from the ascertained coordinates affine transformation has appeared most suitable, whose application make it possible to determine mean deformation of an area covered by a set of points (Fig. 3).

2.3.4. Coefficients of affine transformation, strain and rotation

Affine transformation is described by equations

$$x_i' = a_{ik} x_k'' \qquad i, k = 1, 2$$

in which a_{ik} are 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 used in [11]. In accordance with the basic relations of the theory of deformations subsequently the formula for the tensor of finite strains [12] is obtained

$$e_{ij} = \tfrac{1}{2}(a_{ki} \times a_{kj} - \delta_{ij}) \qquad i, j, k = 1, 2$$

and vector of rotation

$$\omega_{ij} = \tfrac{1}{2}(2a_{ij} - a_{ki} \times a_{kj} - \delta_{ij}).$$

3. Microscopic analysis

3.1. Instruments and equipment

The photographs were taken on a Jeol SEM in the Department of Materials of the Faculty of Nuclear and Physical Engineering of Technical University, Prague. The deformations were produced by a fixture of our own design with a bolt controlled jaw movement. The fixture, together with the inserted sample, was inserted into grooves in the microscope camera. Neither the magnitude of induced deformations nor the force were measured directly. For the analysis of the deformation mechanism a grain of simple shape, well visible boundary and contrasting surface was selected (Fig. 2). The magnification and the grain size were selected in mutual ratio so as to enable the picture to show the grain in sufficient size as well as its considerably large environment.

3.2. Photographing and measurement errors

When using the time base method (Sections 2.1 and 2.2), the fundametal prerequisite for the provision of suitable FGM pictures is the preservation of external and internal orientation elements. In the case of SEM it means the preservation of magnification and geometry of representation in time and the preservation of position of photographed object.

3.2.1. Magnification errors

With regard to the stresses in the fixture producing

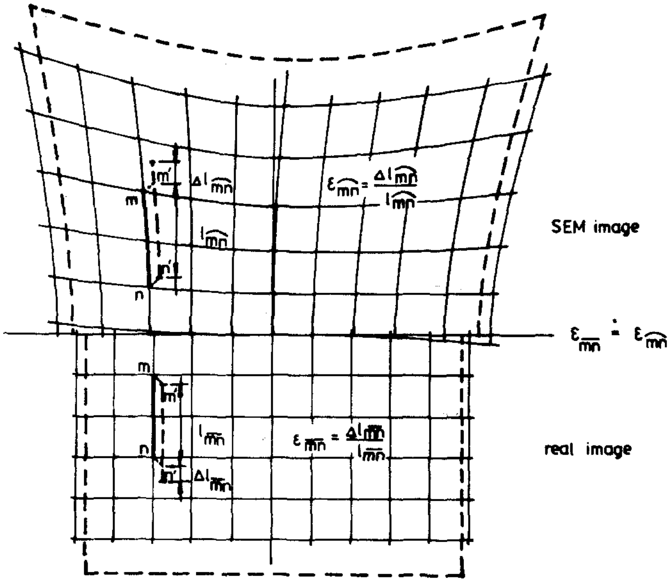

Figure 4 Picture distortion in the SEM and determination of deformations from the photograph.

tensile deformation small changes in the focusing of the microscope and in the position of the sample occur. At every loading step, i.e. when taking the picture preceding and following deformation, the microscope must be refocused, which results also in a certain small deviation in magification, which is of the same sign and magnitude about the whole surface of the picture. This error is most significant, as it manifests itself as strain and influences the calculated mean strain value. In these measurements, it could not be eliminated; it was only possible to estimate the accuracy as a whole in the course of the evaluation of strains. The magnitude of the calculated rotation, however, has not been influenced by this magnitude of deviation, as it is subtracted in the formula for the calculation of vector rotation as a symmetrical part of the transformation coefficients.

3.2.2. Errors in presentation and position of the sample

The errors of presentation depend on the properties of the microscope which must be dealt with in greater detail. As we have already mentioned in Section 2.1.2, a certain distortion of the picture occurs in the SEM, which is shown in Fig. 4. Provided this distortion does not change with time and the pictures made in time base differ from each other only in deformation, then the ratio of the lengths of any line from the pictures taken before and after deformation is constant – with the exception of second order quantities – in any place of the picture field. This is the basis of the possibility for using the SEM pictures made by the time base method for photogrammetric analysis of deformations, which are independent of the geometry of the picture. The change of the picture, similarly as the picture 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 value of strain. Also the errors are of an analogous character, due to the angular change of the sample position. The error due to the change in distance is already included in the error due to the change in magnification. Small rotations of the sample, whether in respect to the axes of the picture or in respect of the optical axis, are centrally antisymmetric and do not influence the mean strain value determined from the pictures. They manifest themselves in the mutual rotation of both pictures, but with regard to the fact that they are incorporated in both the absolute rotation of the grain and in the absolute rotation of its environs, they do not influence the relative rotation of the grain with regard to its neighbours, since they are mutually substracted in the calculation.

4. Analysis of microdeformations of polycrystal aluminium

4.1. Experimental material

The sample used was prepared from the experimental material, aluminium of purity 99.85%,

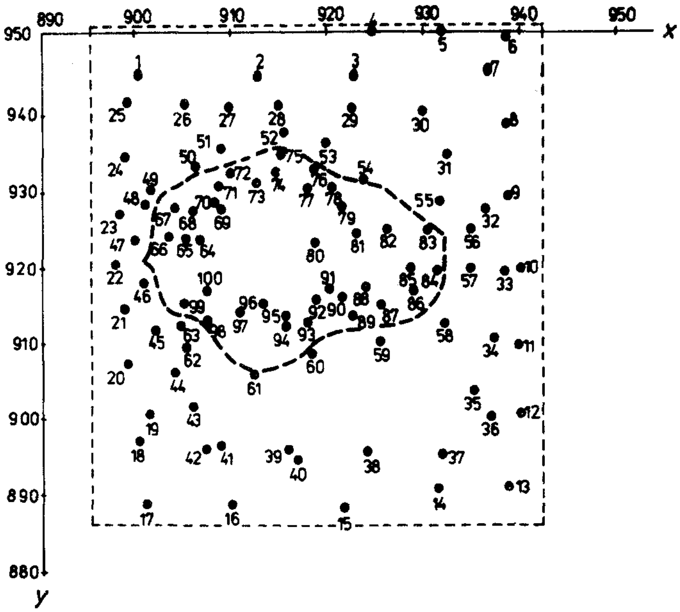

Figure 5 Field of measured points in the photogrammetric pair of photographs.

which had been previously cast into a block 230 mm thick and homogenized for 24 h at a temperature of 580° C. Subsequently it was rolled at a temperature of 420° C into sheets 2 mm thick (80% reduction). By heat treatment at a temperature of 350° C for a period of 4 min internal stresses were eliminated first and by the prolongation of exposure to heat to 4 h samples with a different grain size were obtained by recrystallization. A selected sample was taken from a series which was exposed to heat for 20 min; the mean grain size was 50 μm. The samples were prepared in the Research Institute of Metals in Panenské Břežany. For the purpose of microscopic analysis a sample of the dimensions of 0.5 mm × 7 mm × 30 mm was made of this material. Its thickness was produced from the original 2 mm of the sheet by etching and its surface was polished.

4.2. Description of measured points

In accordance with the conditions which appeared in the stereocomparator as most suitable for the coincidence of the points in photogrammetric pictures taken in the SEM parallaxes were consecutively measured in 100 points, whose distribution about the picture is shown in Fig. 5. With regard to the program of the analysis, described in Section 2.3.4, the points have been divided into two groups, namely, the external points and the internal points. The external points, numbering about 60, are situated outside the grain; the internal points, numbering about 40, are situated inside the grain, whose boundary is shown in the picture. For every group, as well as for the sum of 100 points, the coefficients of an affine transformation, deformation tensor and rotation vector components were calculated.

4.3. Loading process

As we have already mentioned, the magnitude of macrodeformations was controlled only approximately by the turning of the screw of the loading fixture which did not permit an accurate setting of the beginning of loading or the return to a certain position. This circumstance considerably limited the possibility of directly determining the character of the deformation process, and the microdeformations could be observed only in accordance with the increase and decrease of load. The loading diagram (Fig. 6) shows the loading procedure and the points in which the microscopic pictures were taken.

4.4. Evaluation of microdeformations

The process of evaluation of microdeformations from the pairs of photogrammetric pictures has been described in Section 2.3.4. Its practical realization 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

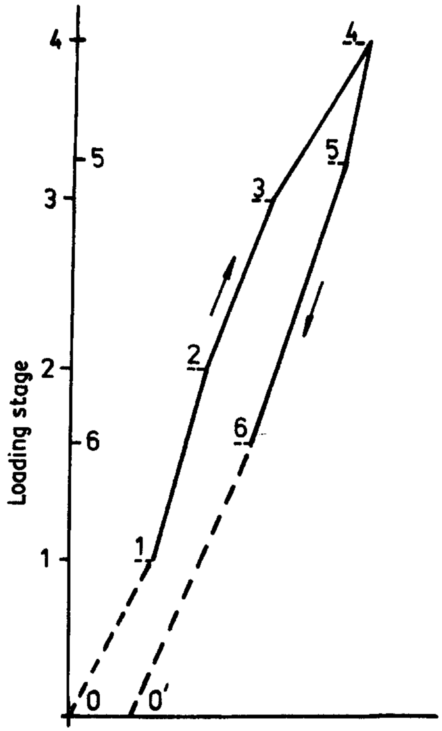

Figure 6 Loading diagram.

components of the strain tensor and rotation vector on a Hewlett Packard calculator (Fig. 7). The results of numerical solution were used for the evaluation of the microdeformation process by two ways.

4.4.1. Strains of the whole picture area

To objectivize the microdeformation process, i.e. to prove agreement between the history of the microstrain and loading in respect of its increase and decrease, the strain tensor components e_{ij}, $i, j = 1, 2$, were calculated from all 100 points distributed over the whole area of the picture in every interval. The results are shown summarized in Table I showing first all strain tensor components, separately for every interval, and, in the next line, the differences Δe_{22} ascertained between the overall strain value in the interval (01 0k) and the sum of partial values. The e_{22} component of strain tensor is highest in all intervals and, consequently, corresponds most in history to the history of the loading process (Fig. 8). No conclusions can be made from the history of other components or the ratio of their values, since they are probably more influenced by local conditions. It is also difficult to discuss absolute values of measured strains because of the lack of data on external forces. However, the differences Δe_{22} according to Table I make it possible to make conclusions about the accuracy of determination of strains with regard to their overall values. Since the pictures have been taken during the same loading cycle and their dimensions cannot be influenced (with the exception of the errors considered), the differences must be considered as the overall error of the microphotogrammetric analysis. Apart from the points on the samples, also four (sometimes even more) points on the frame of the photographs were measured, which are identical in all pictures and independent of the representation and defor-

Interval	Point	x'	y''	p_x	p_y
0103	0053	400 719	921 629	658.987	447 736
0103	0054	406 397	921 701	659 012	447 753
0103	0055	416 296	921 621	659 427	447 754
0103	0056	425 985	919 654	659 406	447 780
0103	0057	426 686	914 856	659 403	448 060
0103	0058	417 969	913 826	659 293	447 958
0103	0059	413 894	916 081	659 072	447 749
0103	0060	406 980	913 689	658 778	447 997
0103	0061	404 167	914 291	658 704	447 895
0103	0062	394 615	915 188	658 445	447 614

(a)

AFFINE TRANSFORM
A1, A2, B1, B2,
1.026 922 451E 00
6.740 642 795E-03
3.507 574 850E-02
9.759 534 903E-01
DEFORMATION
E 11, 22, 12, 21
2.730 757 826E-02
−2.314 223 827E-02
2.129 931 374E-02
2.129 931 374E-02
ROTATION
R 11, 22, 12, 21
−3.851 273 500E-04
−9.042 714 100E-04
1.377 643 476E-02
−1.455 867 095E-02

(b)

Figure 7 (a) Record of stereocomparator measurements. (b) Record of numerical solution values.

TABLE I Values of strain tensor and rotation vector components

Loading		Deformation					Rotation		
Stage	Interval	$\Delta e_{22} \times 10^2$	$e_{11} \times 10^2$	$e_{22} \times 10^2$	$e_{12} \times 10^2$	$e_{21} \times 10^2$	$\Delta\omega_{12} \times 10^2$	$\omega_{12} \times 10^2$	$\omega_{21} \times 10^2$
1	0102	0.131 06	−0.174 14	3.128 75	−1.380 26	−1.380 26	0.181 51	1.232 58	−1.232 58
2	0203	0.332 46	0.000 00	1.844 60	−1.411 44	−1.411 44	−0.179 65	−1.977 02	1.571 82
3	0304	0.541 00	−3.321 30	1.978 83	−1.902 66	−1.902 66	0.23313	1.977 02	−1.977 02
4	0405		2.697 63	−0.833 88	0.032 77	0.032 77		−0.132 38	0.132 38
5	0506		2.868 88	−0.012 08	−1.956 47	−1.956 47		0.019 50	−0.022 40
6									

mation, and are well visible. Their separate evaluation afforded the "strain" magnitudes of about 0.5×10^{-2} which is in good agreement with the deviation Δe_{22}. Therefore, it is possible to conclude that this deviation represents the error of the measured values e_{22} and that in this sense these values are real. The results of deformation analysis of the remaining pictures are not given, since they lay outside the framework of the measurements and are attributed to the uncertainty of the beginning and final loading state.

4.4.2. Grain rotation

The evaluation of the relative grain rotation with regard to its environs is generally analogous with the evaluation of the strains of the whole picture. In every loading interval the overall rotations were calculated in accordance with Section 2.3.4 separately for external and internal points, and from their difference the relative grain rotation, with regard to its environs, was determined. Since the rotation components ω_{11} and ω_{22} are quantities of the second order and are not used in the linear theory of deformation, and hence are not shown in the table. The record (Fig. 7) shows that their values are very small in comparison with the values of the components $\omega_{12} = -\omega_{21}$. The components of rotation ω_{12} and their differences $\Delta\omega_{12}$, determined by the same method as the deviations of strains, are also shown in the table (Table I) and in the diagram (Fig. 8). Their magnitudes in the individual intervals are of the same order as the strain e_{22} and its deviations Δe_{22}, which is considered proof of their objectivity with regard to the changes of load, although the history does not unambiguously correspond with them. However, there are not sufficient data to elucidate this phenomenon, which could be due to both the elastic stability properties of the polycrystalline system [13] and the non-elastic behaviour. The evaluation of pictures 00′ namely yielded residuary relative rotation of the magnitude of $= 1.78 \times 10^{-2}$, whose magnitude is comparable with the magnitudes of relative rotations in the individual

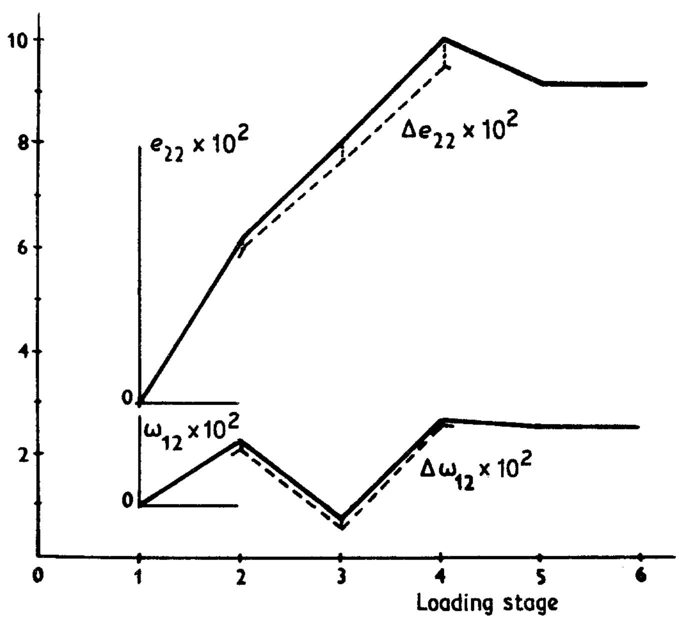

Figure 8 Microstrain diagram of the photograph and diagram of relative rotations of the grain.

intervals, while in the case of strains the magnitude of residual strain e_{22} is comparable with the deviations of their values.

5. Conclusion

The microstrain diagram (Fig. 8) does not make it possible to decide unambiguously about the character of the deformation process, i.e. to determine if the deformations are elastic or elasto-plastic. No proportionality has been ascertained between strains and rotations in intervals with either increasing or with decreasing load. However, the diagram does confirm unambiguously, and with sufficient accuracy, the qualitative agreement between the histories of load and microstrains in the direction of the load applied, both in the region of its increase and decrease. The analysis has confirmed quite unambiguously the relative rotation of the grain in the whole scope of loading. With regard to uncertainty of its character it is impossible to say, whether and when the rotation is reversible or irreversible. However, already the existence of the latter should confirm the presence of the former, similarly this is the case for strains. The relatively small rotation and the relatively small strain under first load release have also the sense identical with the history of the process and indicate that elastic rotation can be expected in this phase. As the measurements have not been always entirely compatible with respect to the preservation of the elements of internal and external orientation in the taking of photogrammetric photos, the required accuracy in the calculated values of strains and rotations has not been attained, either.

With regard to the fact that it is possible to improve the technical conditions for the taking of photogrammetric photographs and for the coincidence of the points in the pictures in the stereocomparator, it is also possible to approach closely the theoretical accuracy of these measurements which is deduced from the internal accuracy of the stereocomparator.

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 microdeformations of material structures.

Acknowledgement

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Appendix: Affine transformation [11]

If the presentation is defined as

$$x_i'' = a_{ik} x_k' \qquad \text{(A1)}$$

and for a set of points N both their coordinates $x_i'(N)$ and their representations $x_k''(N)$ are known, the coefficients of representation a_{ik} are determined in accordance with the following algorithm.

First the coordinates of the points are converted with regard to the centroid of the whole set. Since all points have the same weight, it is considered as equal one, and the coordinates of the centroid follow out of the relations

$$x_{0i}' = \frac{\sum^N x_i'(N)}{N} \qquad x_{0k}'' = \frac{\sum^N x_k''(N)}{N} \qquad \text{(A2)}$$

and the reduced coordinates are determined from the differences

$$X_i' = x_i' - x_{0i}' \qquad X_k'' = x_k'' - x_{0k}''. \qquad \text{(A3)}$$

After the development of transformations Relation A1 for reduced coordinates X_i' and X_k'' we obtain equations

$$\begin{aligned} X_1'' &= a_{11} X_1' + a_{12} X_2' \\ X_2'' &= a_{21} X_1' + a_{22} X_2' \end{aligned} \qquad \text{(A4)}$$

whose coefficients are determined from the terms

$$a_{11} = \frac{AG - BD}{J} \qquad a_{12} = \frac{ED - BG}{J}$$

$$a_{21} = \frac{AF - BC}{J} \qquad a_{22}^2 = \frac{EC - BF}{J} \qquad \text{(A5)}$$

where

$$A = \sum^N X_2''(N) \times X_2''(N) \qquad E = \sum^N X_1''(N) \times X_1''(N)$$

$$B = \sum^N X_2''(N) \times X_1''(N) \qquad F = \sum^N X_1''(N) \times X_2'(N)$$

$$C = \sum^N X_2''(N) \times X_2'(N) \qquad G = \sum^N X_1''(N) \times X_1'(N)$$

$$D = \sum^N X_2''(N) \times X_1'(N) \qquad J = AE - BB \qquad \text{(A6)}$$

References

1. B. M. ROVINSKIJ and V. M. SINAJSKIJ, "Nekotorye problemy prochnosti tverdogo tela" ("Some problems of strength of solids") (USSR Acad. Press, Moscow, 1958).
2. T. H. DAWSON, PhD thesis, The Johns Hopkins University, Baltimore, Maryland (1968).

3. L. BERKA, *J. Mater. Sci.* **17** (1982) 1508.
4. A. BOYD, *J. Microsc.* **98** August (1973).
5. K. SCHWIDEFSKY, "Grundriss der Photogrammetrie" (B.G. Teubner-Verlag, Stuttgart, 1963).
6. A. BOYD and H. F. ROSS, Photogrammetric Record 8 (46) (1975).
7. Proceeding of the XIII Congress of the International Society for Photogrammetry, Commission V, Helsinki, 1976 (published by Secretariat of the Congress, Helsinki).
8. Proceeding of the XIV Congress of the International Society for Photogrammetry, Commission V, Hamburg, 1980 (Inst. of Photogrammetry, University of Bonn, 1980).
9. K. B. ATKINSON, *Photogram. Eng.* **42** (1976) 1.
10. P. NOVÁK, "Application of Stereofotogrammetry in Deformation and Stress Analysis" (Rozpravy ČSAV, Řada techn. věd., 1967) roč. 77, seš. 5. (In Czech).
11. W. JORDAN, O. EGGERT and M. KNEISEL, "Handbuch der Vermessungskunde" Vol. 2, S.83 (Stuttgart, 1963).
12. W. NOWACKI, "Teorija sprezystošci" (Pan. Wyd. Naukowe, Warsaw, 1970).
13. J. F. BELL, "The Physics of Large Deformation of Crystalline Solids", Springer Tracts in Natural Philosophy, Vol. 14, (Springer-Verlag, Berlin, 1968).

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