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Characterization of Biocompatible Materials Using Stereo Microscope 3D Digital Image Correlation**

By *Tomasz Rusin* and *Magdalena Kopernik**

This paper presents the measurement of the sub-micrometer deformation of biocompatible materials induced by mechanical and thermal loading with a newly developed stereoscopic microscope 3D digital image correlation (DIC) system. This technique combines a 3D DIC system based on two cameras in a stereoscopic setup with a stereoscopic microscope. This paper describes measurements of displacements in the range of 100 microns with an error of 0.1%, which is several times better than the accuracy provided by other comparable systems. The examined micro-sized specimens are cut out of biocompatible material used for the construction of a pneumatic ventricular assist device. The results include a 3D surface profile, analysis of the sample surface deformations, and distribution of surface strains.

1. Introduction

The two-dimensional digital image correlation (2D DIC) technique was proposed in the 1980s for measuring surface displacement (in-plane 2D measurements).[1,2] The 2D method was successfully used for measurement of the mechanical properties of biological tissues.[3] In the past 20 years, this method has been improved in many respects,[4–7] including measurement accuracy, high-speed digital imaging cameras, automated calculation and visualization modules for stresses and strains, extended data exporting, and reporting options. The 3D DIC technique is currently standardized and widely applied in experimental mechanics and structural analysis.[8–10]

Recent progress in the digital image correlation method includes introduction of 3D systems to microscopes,[10–13] multiple camera DIC,[14] real-time measurement (real-time image correlation),[15] and validation of the finite element method (FEM) models by DIC measurement results through the integration of FEM and DIC programs. It is also possible to identify the parameters of the rheological equations of materials by coupling DIC systems with devices for mechanical testing and by DIC measurement of specimens during heating/cooling; for example, in order to measure coefficients of thermal expansion.

The main advantage of a 3D DIC in comparison with a 2D system is that in real life most materials deform in all three dimensions (X, Y, and Z). Unfortunately for micromechanical tests, there are not many available solutions which would enable 3D DIC measurement in a range of measurement capabilities described in the present paper. The diffraction grating technique (e.g., Moiré and Shadow Moiré) does not have sufficient spatial resolution to measure submicron displacement of micro specimens.[10] The results produced in the present paper and error estimation section show that the presented micro DIC system enables measurement of displacement and strain for micro-size samples with an accuracy level not met when using either the diffraction grating technique (e.g., Moiré and Shadow Moiré) or many custom-made 3D DIC prototypes based on two cameras in a stereoscopic setup. Most existing 3D DIC systems based on two cameras in a stereoscopic setup do not enable measurements of very small specimens which are smaller than 1 cm^2 with sufficient resolution. Some authors propose prototype 3D stereo microscope DIC systems to measure micro specimens, but these also fail to measure submicron displacements.[11,12] The system used in this study has some special features such as a large depth of field, a large angle between cameras, and an objective with a long working distance which are vital in order to obtain accurate 3D displacement and strain measurement results in micromechanical tests. The system is also vibration resistant. This paper presents measurements of displacements in the range of 100 microns

[*] *M. Kopernik*
AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland
E-mail: kopernik@agh.edu.pl
Dr. T. Rusin
Dantec Dynamics GmbH, Kaessbohrer Str. 18, D 89077 Ulm, Germany

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with an error of 0.1%, which is several times better than the accuracy provided by other comparable systems. Examples of errors reached by other authors include the following:

1) fluorescent stereo microscopy – X – displacement 100 nm measured with an error of 300%; X – displacement 120 μm measured with an error of 0.25%.[13]
2) 3D DIC – horizontal displacement 300 nm with an error of 2 600%; vertical displacement 500 nm with an error of 640%.[12]
3) 3D DIC – components of strain tensor in range of 200–300 measured with an error of 120–150%.[11]
4) 3D DIC – Z – displacement 100 μm measured with an error of 1.3%.[10]

This means that the system used in the present paper based on a stereoscopic microscope with two cameras in a stereoscopic setup could be applied in microscale material research by combining the advantages of a stereoscopic microscope with a 3D DIC system. The presented system can compete with the fluorescent stereo microscopic DIC technique,[13] which has some advantages in the measurement of biological tissues. However, fluorescent stereo microscopic DIC cannot be used for all types of samples. Fluorescence DIC also has some disadvantages related to the duration of the experiment, because fluorescence is not permanent, the samples photobleach, and the fluorescence fades.[16] Therefore, the preparation of samples made of non-biological material tested in a prolonged mechanical experiments is highly unrecommended for fluorescence microscopy. This system requires the use of a fluorescent light source, filters, and a fluorescent speckle pattern. The system described in the present study is especially useful for transparent or semi-transparent materials. Thin foil is an example of such a type of biocompatible material examined in the present paper. Non-crystalline specimens like those analyzed in the present paper cannot be examined by X-ray microtomography and profilometric techniques do not offer sufficient spatial resolution.

In this paper, we present the measurement of sub-micrometer displacement during mechanical characterization of biocompatible materials by applying a newly developed stereoscopic microscope 3D digital image correlation system. The goal of the study is to present the potential of the micro DIC method. Originally, this system was developed for the measurement of 3D warpage and the coefficient of thermal expansion of electronic components in the electronics industry. The examined micro-size specimens were cut out of biocompatible material used for the production of pneumatic ventricular assist devices. The results include presentation of 3D surface profiles, deformation of the sample surface, strains on the sample surface induced by heating and stretching. The measured changes of thermomechanical properties are induced by changes of temperature from room temperature to body temperature, by deposition of nano-coatings on the surface of polymer samples and stretching. The results obtained by DIC were verified by other research methods (finite element method, in situ SEM microshear test, profilometric test) and the high level of consistency of data obtained in the present study.

The detailed DIC deformation analysis of a blood chamber of a ventricular assist device (VAD), with a discussion of measurement errors and error sources was presented previously.[17,18] The blood chambers of VADs (Religa Heart_Ext and Polvad_Ext) were tested by DIC,[17,18] with a unique experimental setup using a physical model of the circulatory system (Windkessel model) to reproduce short- and long-term dynamic operation of the pneumatic ventricular assist device.[19] These measurements were performed on large scale structures (whole VADs were measured). The influence of nanocoating on the mechanical behavior of the blood chambers of a VAD was observed by the authors of the present paper using the DIC method on the VADs.[17] These results encouraged the authors to measure the influence of surface modification (nanocoating) on micro specimens with much lower thickness (thin foil). Subtle differences in mechanical response among different types of prototypes of VADs were also observed by the authors of the present study in their earlier studies using the DIC method.[18] The promising results produced by DIC measurement performed on macro size specimens (real size VADs) with low values of errors and comparable results from numerical models of the VADs encouraged the authors to continue research with the DIC method.[18] The high level of accuracy and high resolution of measurement results obtained by stereoscopic DIC enable validation of mechanical properties calculated by the numerical method (finite element method) to be performed on a micro scale. The results obtained in the present manuscript will be helpful to improve the design, material selection, surface modification, and quality of VADs.

2. Materials and Methods

2.1. 3D Stereomicroscope DIC Method and Experimental Section

Two cameras in a stereoscopic setup connected to a stereoscopic microscope create a unique opportunity to perform 3D deformation and strain measurement of micro specimens. Each camera provides a 2D view. Software forms a 3D image combining (through a correlation algorithm) both camera views from different angles.[20] In the presented system, the angle between the cameras was 17°. In order to make measurements, specimens have to be marked by applying a stochastic pattern to the surface. The best method to create this pattern is to apply a white matte background (by using white matte paint) and after drying to apply black speckles by spraying the surface with black matte (non-reflective) paint. It is also possible to apply dry paints by application of powdered titanium oxide (white) and carbon or magnesium powder (black). One can use particles of different sizes in order to create an optimal speckle size. This stochastic gray value pattern – a unique recognition pattern – can be considered a sort of fingerprint on the surface. In some

materials, it is possible to use a natural speckle pattern (for example, grains of metals). Pattern adhesion is an important factor of DIC accuracy. However, the currently available choice and quality of paints ensures sufficient pattern adhesion. For thin foils and other delicate materials for speckle pattern generation, it is recommended to use water-based chalk spray or dry pigments which can be applied by an airbrush. The advantage of using dry pigments is that they do not contain any binders (solvents) which could chemically modify the surface and they do not create additional film on the surface which could change mechanical properties of tested material. Another advantage of using dry pigments for pattern generation is that it is possible to use particles of specified size range which will be optimal for pattern recognition.

From a stereoscopic perspective, a correlation algorithm needs to identify the same object points – homolog points.[8] The unambiguous gray value pattern on the surface (a stochastic structure) enables the correlation algorithm to calculate 3D coordinates (X, Y, and Z). The transformation correlation algorithm calculates translation, stretching, shear, and distortion. A digital image of the surface can be reconstructed with knowledge of the imaging setup from an intersection of view directions. The first step is the recognition of shape and reconstruction of the 3D surface. In the second step, deformation is measured. In order to measure the deformation, the correlation algorithm compares the reference image with the image acquired after deformation of the specimen (Figure 1a and b). The correlation algorithm does not analyze single points (pixels), but small areas, sub-frames, or facets composed of about 10–30 pixels in length (the length of the rectangle which creates the frame/facet). In order to work properly, the algorithm needs a unique and recognizable pattern in each facet. Facets are not shifted in 2D. Therefore, a simple 2D correlation algorithm is not appropriate. Speckle pattern quality (size of speckles and contrast), size of facets, as well as grid overlap have a strong influence on the accuracy and resolution of measurement (Figure 1c).[9] The measurement error obtained in the present paper is very low and fixed in a narrow range. Therefore, it can be stated that the potential disadvantages of the method have been overcome and the accuracy of measurement has been assessed at a satisfactory level.

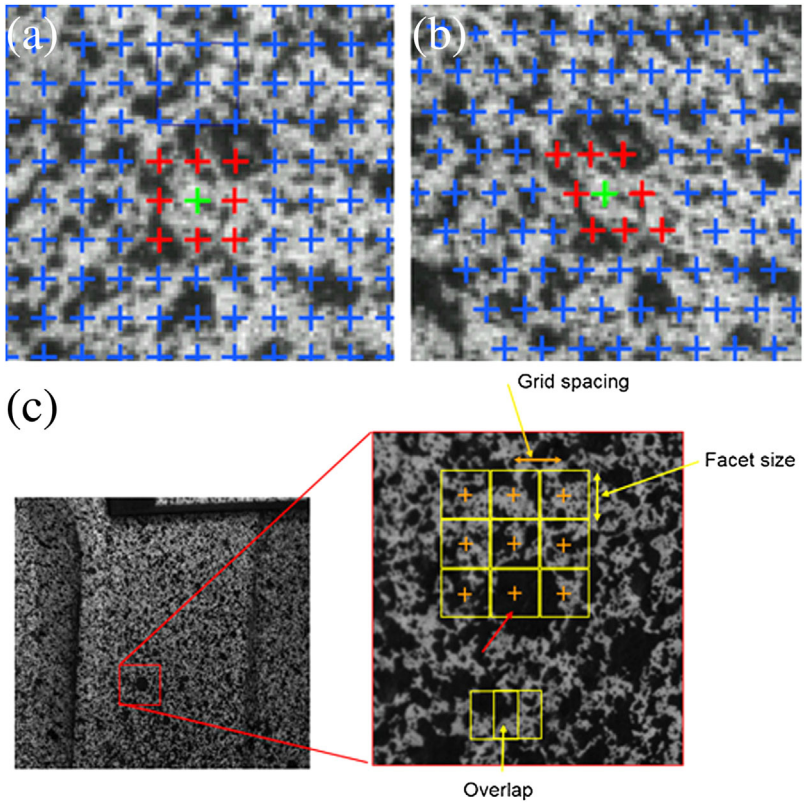

Fig. 1. Recognition of displacement based on an analysis of pixels in the area of facets. (a) Reference image and (b) image of deformed specimen (evaluated state). (c) Facet size, grid spacing, and overlap have to be properly selected in order to accurately calculate specimen deformation and strain. Extensive overlapping leads to artificial smoothing. Larger facets give more accurate results, but with lower resolution.

The correlation algorithm can accurately calculate 3D deformation and strain only when an image from two cameras is calibrated. The calibration process enables the algorithm to correct errors generated by a distortion of an image through the optics of the system (e.g., objective aberration). During calibration, a known pattern (certified calibration board) is imaged under different perspective views. The calibration process enables the calculation of parameters such as focal length, principle point, radial distortion, tangential distortions which are influenced by focal lens, focal distance, and aperture. It also enables the calculation of the angle and distance between cameras in order to create a global coordinate system.[21] Additionally, the calibration process has been standardized, which provides higher confidence in the optical measurement of strain.[8,9] After calibration and measurement, it is possible to calculate 3D displacements and strains. The data are represented in the form of a false color map where different values of strain and displacement are shown in a color scale (Figure 6b and c; Figure 7b and c).

The results presented in this paper show 3D results obtained with a stereoscopic microscope and calibration procedure, without the need for extra distortion correction.[8,9,21]

The 3D stereomicroscope DIC system (model Q–400 μDIC, Dantec Dynamics GmbH, Germany) is used to perform experiments presented in this paper and it is composed of the following:

1) Stereomicroscope with magnification range: 8× – 100× (Planachromatic 1.0× objective, working distance 61.5 mm, 10× eyepieces), apochromatic corrected optics, integrated iris diaphragm for adjustable depth of field, click stop zoom settings for parfocal plan apochromat objectives: 0.63×, 1×, 1.6×, 2×, integrated light-emitting diode (LED)

ring light, gliding stage, etc. Resolution and focal length can be adapted to the individual application. A gliding stage is used for precise positioning of the specimen in the field of view. A special adapter was used which enabled the attachment of two cameras at an angle of 17° in order to make 3D observations of specimens (stereoscopic setup of cameras). This relatively high angle between two cameras gives more accurate out-of-plane (Z axis) measurement results.

2) Two cameras with chip resolution 5 MPx, shutter speed 47 µs – 67 s, frame rate 5 Hz, and adjustable region of interest.
3) Available measurement area – $17 \times 17\,mm^2$ and smaller, and calibration plates ranging from $0.5 \times 0.5\,mm^2$ up to $20 \times 20\,mm^2$. Measurement range is up to several – 100% strain. Measuring sensitivity: displacement is up to 0.01 pixel accuracy depending on measuring conditions and strain is up to 0.01% local accuracy. Measurement results are obtained in a full-field contour as 3D displacements and strains.

Software functionality of the 3D stereomicroscope DIC system includes the following:

1) Real-time correlation up to 5 Hz, a fast and easy automated calibration procedure with accuracy feedback, selectable filtering of measurement data, and free definition of reference step. Unrestricted, open source data format of measurement, extended export, and import functionality (e.g., STL, HDF5, AVI, ASCII, and JPG). Measured data can be extracted as an analog output (±10 V) for closed loop control of external devices such as actuators or temperature chambers.
2) Results displayed on a 3D model and confidence interval for every data point, enhanced external and internal triggering functions. Easy post-processing of data, comfortable visualization, and analysis of dynamic processes, as well as different coordinate systems are available – additionally, a user-definable axis system can be used.
3) Removal of rigid body movement from computed displacements and strains.

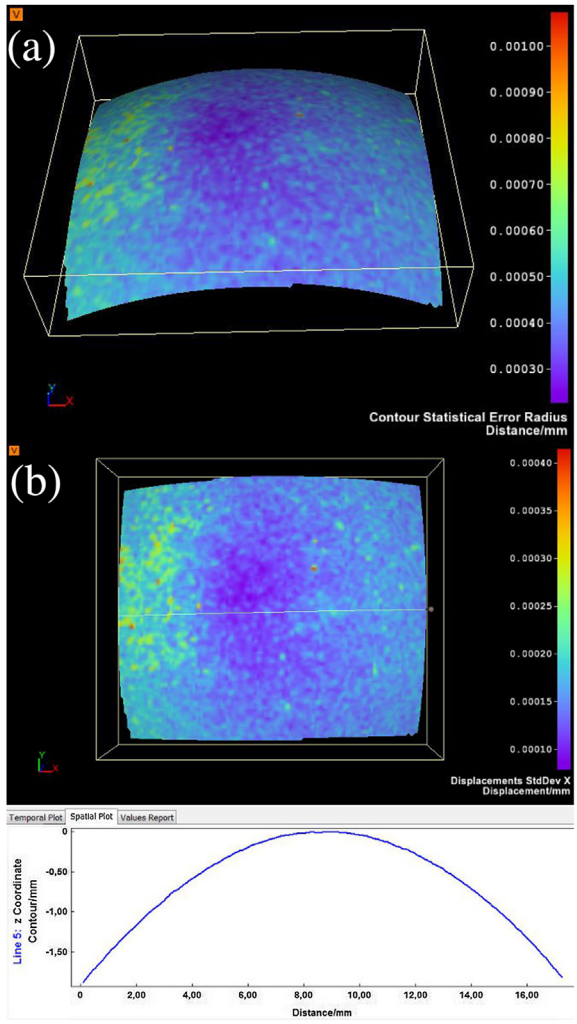

Fig. 2. (a) Distribution of measurement error of the contour of a table tennis ball. (b) Distribution of confidence interval of Z displacement of a table tennis ball. The Z coordinates are presented for the marked measurement line visible on the picture above.

The described 3D stereomicroscope DIC was designed for the measurement of 3D warpage and thermal expansion for micro-electronic/components used in the electronics industry. The presented hardware and software configurations can precisely measure 3D deformation. The authors of this study decided to test the applicability of a stereomicroscope 3D DIC system on micro-sized samples made of biocompatible materials other than electronics, where accuracy is also required at the micro level. The existing setup enables the loading of micro-specimens not only by thermal load, but also by mechanical load, as well as combined thermal and mechanical loads. Mechanical load can be applied by micro-actuators (e.g., step

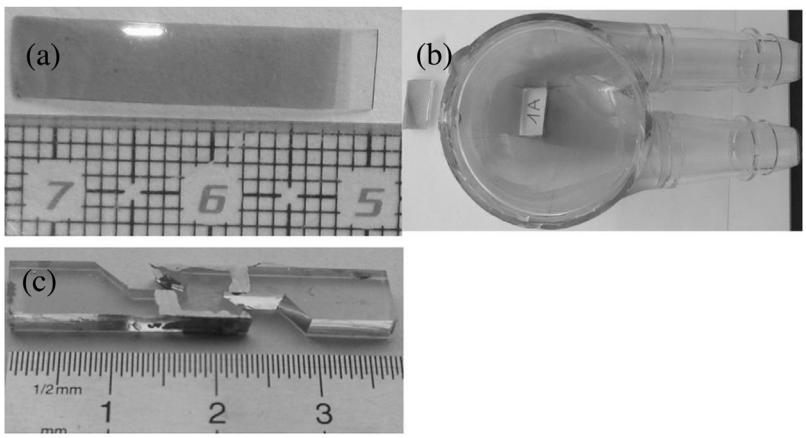

Fig. 3. Photos of samples tested in the 3D stereomicroscope DIC system: (a) thin foil, (b) inner part of blood chamber of VAD, and (c) shear test sample.

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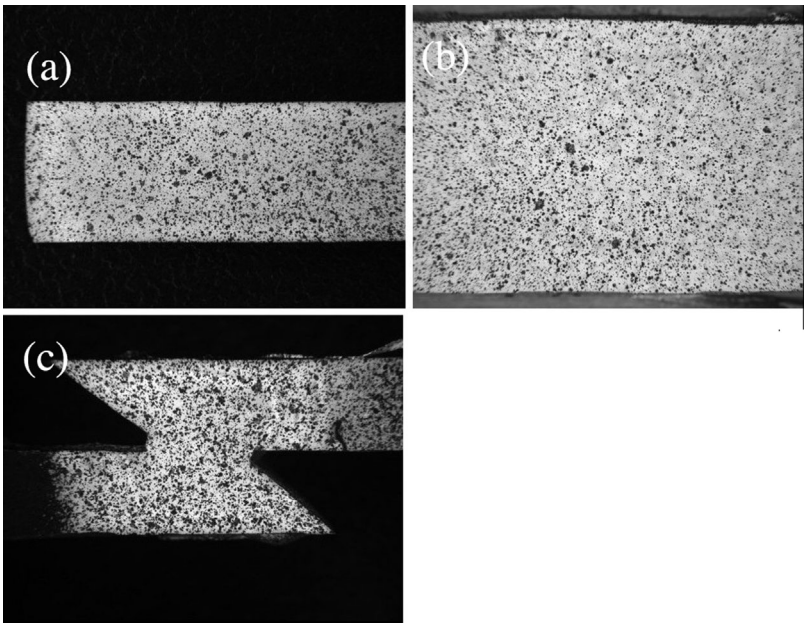

Fig. 4. View of flat samples with paint speckles used in the 3D stereomicroscope DIC system: (a) thin foil – sample prepared for measurement of residual stress, (b) sample cut out from blood chamber of VAD and heated, and (c) sample stretched in shear test.

motors and piezo actuators), micrometers, and other mechanical devices capable of applying mechanical load in a controlled fashion.

2.2. Error Estimation of a 3D Stereomicroscope DIC System

Measurement uncertainty and sources of errors from the 3D stereomicroscope DIC technique and calculation algorithm, as well as a full description of error estimation can be found in separate papers.[18,21] Since a correlation algorithm always compares pictures of two different deformation stages, which can be induced by heat or mechanical load, it is possible to measure the error of calculation algorithm by comparing values calculated for the same deformation stages. The best method for verifying the displacement measurement error and accuracy of 3D DIC is to measure the displacement of a speckle pattern placed on a certified displacement calibrator (used for calibrating calipers and extensometers). The measurement errors (confidence intervals) of displacement were always in the sub-micrometer range. The values are presented below in their respective figures and tables.

The uncertainty of the reconstructed position can be estimated for every calculated point on the object. Therefore, the contour/error radius is introduced as the radius of a sphere around the calculated position in which the right/correct position is expected to reach the probability of ≈67%. This describes the accuracy of a reconstructed point in 3D. The measurement uncertainty is calculated for each data point independently, provided that every point is calculated independently. The error estimation is not valid if the data points are not independent.

The contour of a table tennis ball was measured by the 3D stereomicroscope DIC system to verify the accuracy and depth of field in the present study. The maximum area of the view field was $17 \times 14\,mm^2$. This was measured by the system using 10 000 points and the coordinates were calculated for all the points. The measurement error of the contour (Figure 2a) was less than 100 nm (geometry measurement – data comparable to profilometer data). In contrast, the wavelength of visible light is 300–700 nm. The confidence interval of *Z*

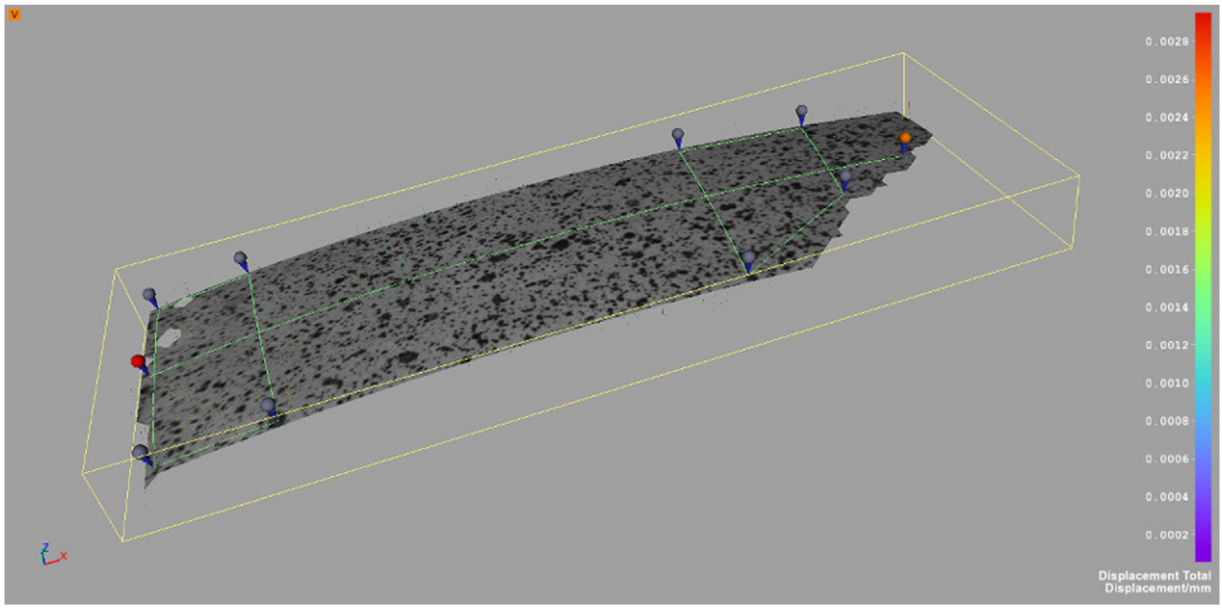

Fig. 5. 3D contour view of thin foil with marked areas, lines, and measurement points.

displacement (Figure 2b) was higher, but it did not exceed 0.00028 μm. The highest error is observed at the edges of the sample, but this is caused by the laws of optics. There is a lack of reference data at the edge which refers to the results just behind the edge. Thus, the algorithm calculates the value on the edge and only uses several pixels (3–5 pixels) to calculate a single facet.

2.3. Advantages of a 3D Stereomicroscope DIC System and Comparison with Other Optical Methods of Measuring Deformation in Micro Scale

The deformation measurement in a micro and lower scale using optical methods can be done using the following: i) the 3D SEM Moiré method (SMM) for 3D shape measurement with nano-scale sensitivity;[22] ii) the micro-lattice method based upon deformed transmission-virtual grating for determination of the micro 3-D surface of deformed liquid with a submicron scale resolution;[23] iii) the microelectronic speckle pattern interferometry (ESPI) system for tensile stress–strain curves measurement;[24] iv) and ESPI can be used during the hole drilling method for determining macroscopic near surface residual stresses.[25] The accuracy of the above methods depends upon the accuracy of the cameras and sensors used, and in some cases these methods can be applied interchangeably.

Moire methods require a surface preparation. DIC also requires a surface preparation (pattern). However, sample preparation SMM poses greater difficulties than in the 3D stereomicroscope DIC method. Besides, samples used in this study are translucent, which precludes their usage in the other above optical methods. Moire and ESPI techniques have a limitation in dynamics per measurement and require multiple images per measurement step. The DIC technique only needs a pair of images at each loading step, and measures contours and displacement directly. The 3D stereomicroscope DIC method enables quicker loading steps (thermal and mechanical) in comparison with ESPI. 3D stereomicroscope DIC is less sensitive to vibration, so it can be used with different material testing machines (electrodynamic, servohydraulic, and electromechanical). ESPI (based on the interferometric principle) is very sensitive to vibration. Therefore, it is necessary to eliminate any source of mechanical noise in order to perform mechanical tests. However, mechanical noise is sometimes very difficult to eliminate. It is possible to apply the quick loading method (mechanical and thermal) with 3D stereomicroscope DIC. The ESPI method requires small

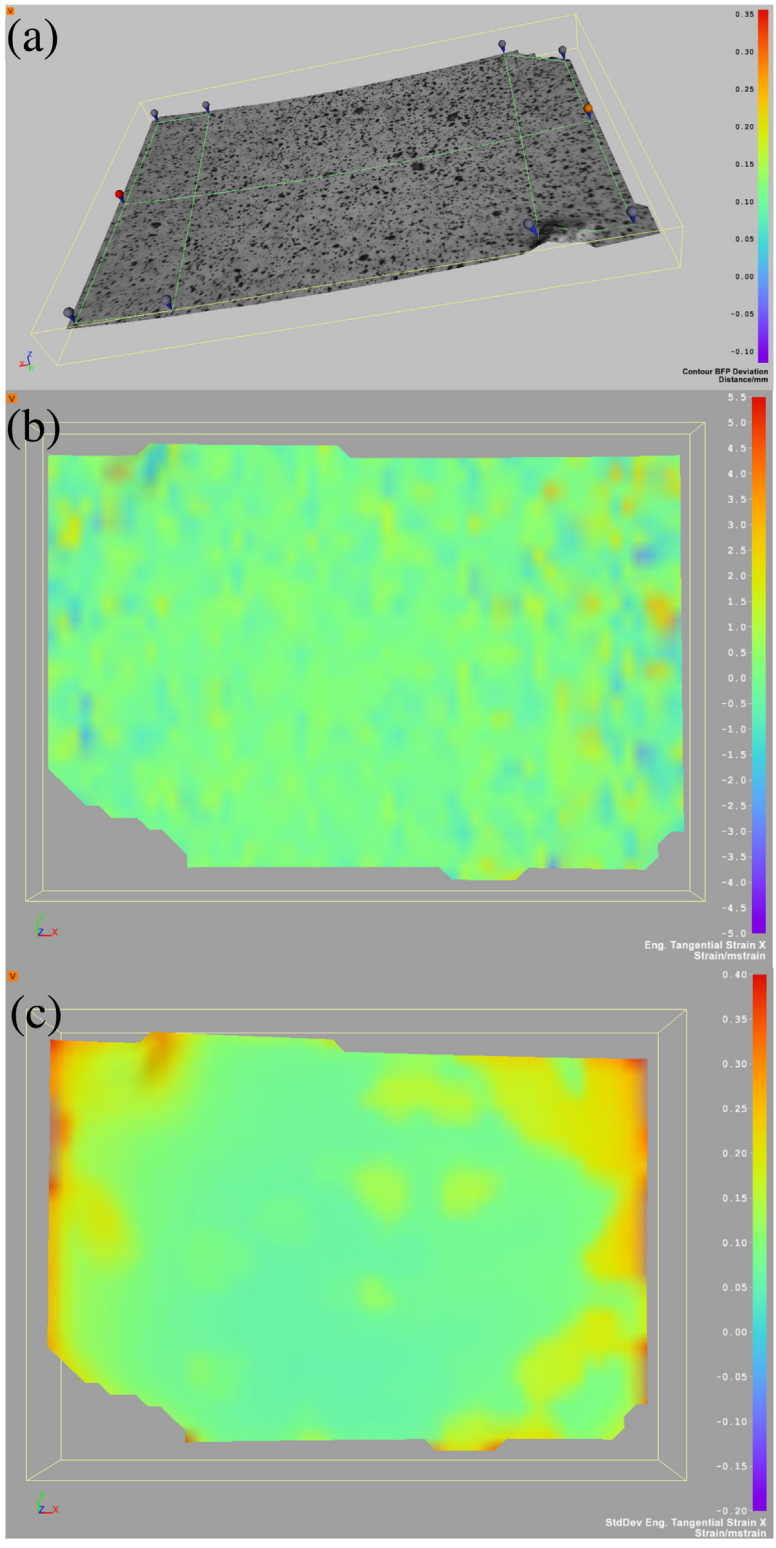

Fig. 6. (a) 3D contour view of heated sample with marked areas, lines, and measuring points. (b) Distribution of ε_{xx} computed in a 3D stereomicroscope DIC system for a sample made of biocompatible materials of a blood chamber of ventricular assist device treated at a temperature of 38 °C for 15 min. (c) Distribution of the confidence interval of ε_{xx} computed by 3D stereomicroscope DIC software for a sample made of biocompatible materials of a blood chamber of a ventricular assist device treated at a temperature of 38 °C for 15 min.

steps and longer periods of stabilization to obtain images which are not "blurred" by noise. 3D stereomicroscope DIC is capable of measuring plastic deformation of specimens up to 300% and more. Another advantage of 3D stereomicroscope DIC is that it is very easy to connect micro DIC to existing material testing machines due to the large working distance of the stereomicroscope objective. The long depth of focus enables the analysis of larger areas and objects of higher curvatures – which is especially useful during measurements of bones, dental implants, tissues, etc.

The main challenge in the development of the stereomicroscope used in 3D DIC was to adapt the pinhole model to that type of imaging system (a stereoscopic microscope uses pseudoscopical imaging) which enabled the calibration process of this optical instrument. Another challenge was to obtain a high angle between two cameras connected to the single objective of a stereoscopic microscope.

2.4. Sample Preparation and Test Conditions

The capabilities of the 3D stereomicroscope DIC system were verified in tests carried out on flat micro-size samples made of biocompatible materials applied in pneumatic ventricular assist devices.[26] Polymer samples were manufactured as standardized (EN ISO 294–2) samples made of Bionate II 90A. The standardized samples were manufactured by an injection molding process. Items of pulsatile VAD (Religa Heart_Ext model) were manufactured according to an industrial process developed by the Foundation of Cardiac Surgery Development (FRK) in Poland.[26] The VAD consists of blood and pneumatic chambers, and inlet and outlet connectors. The specified items are made of the Bionate II 90A by injection molding. TiN coatings were deposited on the Bionate II substrates using a pulsed Nd:YAG laser system. The parameters of the deposition process were determined at the Faculty of Metal Engineering and Industrial Computer Science of the AGH University of Science and Technology in Poland.[27] The parameters of the deposition process were as follows: energy of a laser beam (100 mJ), wavelength (266 nm), fluence (4.2 J cm^{-2}), temperature of the substrate (25 °C), pulse duration (12 ns) at a repetition rate of 10 Hz, and 5 000 laser shots. The gold was deposited by a magnetron sputtering method with a discharge current of 10 mA and a deposition time of 5 min.

The first of the analyzed micro-size samples in the 3D stereomicroscope DIC system was a thin film composed of 50 nm TiN, 5 nm gold, and 0.14 mm Bionate II (Figure 3a). A view of the painted half of the sample is shown in Figure 4a. The dimensions of the tested specimens were as follows: thickness 0.14 mm, width 5 mm, and length 23 mm. The introduction of the gold interlayer to the material system allowed the application of an alternative method of residual stress measurement – the profilometric test.[18] The interlayer of gold increased compressive stresses in the material system and caused the deflection of the entire sample. For this purpose, the residual stress calculations were conducted with Timoshenko's analytical model on the basis of the results of profilometric tests.[28,29] The calculated value of residual stress in the TiN coating was 690 MPa. Measurement of the deflection of the sample in this study was carried out using the 3D stereomicroscope DIC system instead of the profilometer.

The second of the coated polymer samples tested in the 3D stereomicroscope DIC system was cut out from the inner part of the blood chamber of a pneumatic VAD (Figure 3b). A view of the surface of this sample with a speckled pattern is shown in Figure 4b. The examined objects were Religa Heart_EXT pumps not equipped with valves. The pumps were made of biocompatible polyurethanes according to a technological process developed by the Laboratory of Artificial Heart of the Foundation of Cardiac Surgery Development in Poland.[26] The inner surfaces of the polymer blood chambers were modified by coatings: 35 nm of TiN and 5 nm of Au. The dimensions of the tested specimens were as follows: thickness 3 mm, width 10 mm, and length 20 mm. The sample was heat loaded from 24 to 38 °C. During this process, photos were taken at every degree. The highest temperature was maintained for 15 min, after which time the last photo was taken.

The last sample was prepared for a shear test (Figure 3c) using the 3D stereomicroscope DIC system and a view of the surface of this sample with a speckled pattern is shown in Figure 4c. The sample was made of Bionate II coated with 35 nm of TiN and 5 nm of Au. The dimensions of the tested specimens were as follows: thickness 3 mm, width 10 mm, and length 55 mm. The average measured distance between two points in the center of each specimen was 5 mm. During the shear test, the sample was elongated in steps by a micrometric screw in the range from 0 to 0.748 mm. After each step of elongation, a photo was taken.

3. Results and Discussion

The contour view of the first tested sample after processing by the micro DIC software with marked measurement points and measurement line is shown in Figure 5. The deflection measured with the profilometer is 0.976 mm and

Table 1. Averaged components of a strain tensor in mm m^{-1} and their accuracy in mm m^{-1} for line and two contours of a sample heated to selected temperatures: 24, 32, and 38 °C.

Temperature		24 °C	32 °C	38 °C
Line	ε_{xx}	0.00 ± 0.13	0.15 ± 0.13	1.74 ± 0.13
	ε_{yy}	0.05 ± 0.04	0.23 ± 0.04	2.22 ± 0.04
	ε_{xy}	0.03 ± 0.04	0.05 ± 0.04	0.01 ± 0.04
	ε_{xx}	0.00 ± 0.01	0.13 ± 0.10	1.63 ± 0.10
Contour 1	ε_{yy}	0.02 ± 0.03	0.18 ± 0.03	2.14 ± 0.03
	ε_{xy}	0.00 ± 0.03	0.02 ± 0.03	0.04 ± 0.03
	ε_{xx}	−0.01 ± 0.12	0.10 ± 0.12	1.63 ± 0.12
Contour 2	ε_{yy}	0.01 ± 0.04	0.19 ± 0.04	2.23 ± 0.04
	ε_{xy}	0.01 ± 0.04	0.01 ± 0.04	−0.11 ± 0.04

for the same sample the deflection measured using the 3D stereomicroscope DIC method is 1.2 mm.[18] The difference in the measured values is caused by the choice of the measurement points. The tested 3D stereomicroscope DIC method can be used interchangeably with the profilometric test, providing similar results and a similar accuracy of results (accuracy to thousandths of a millimeter is achieved in both methods).

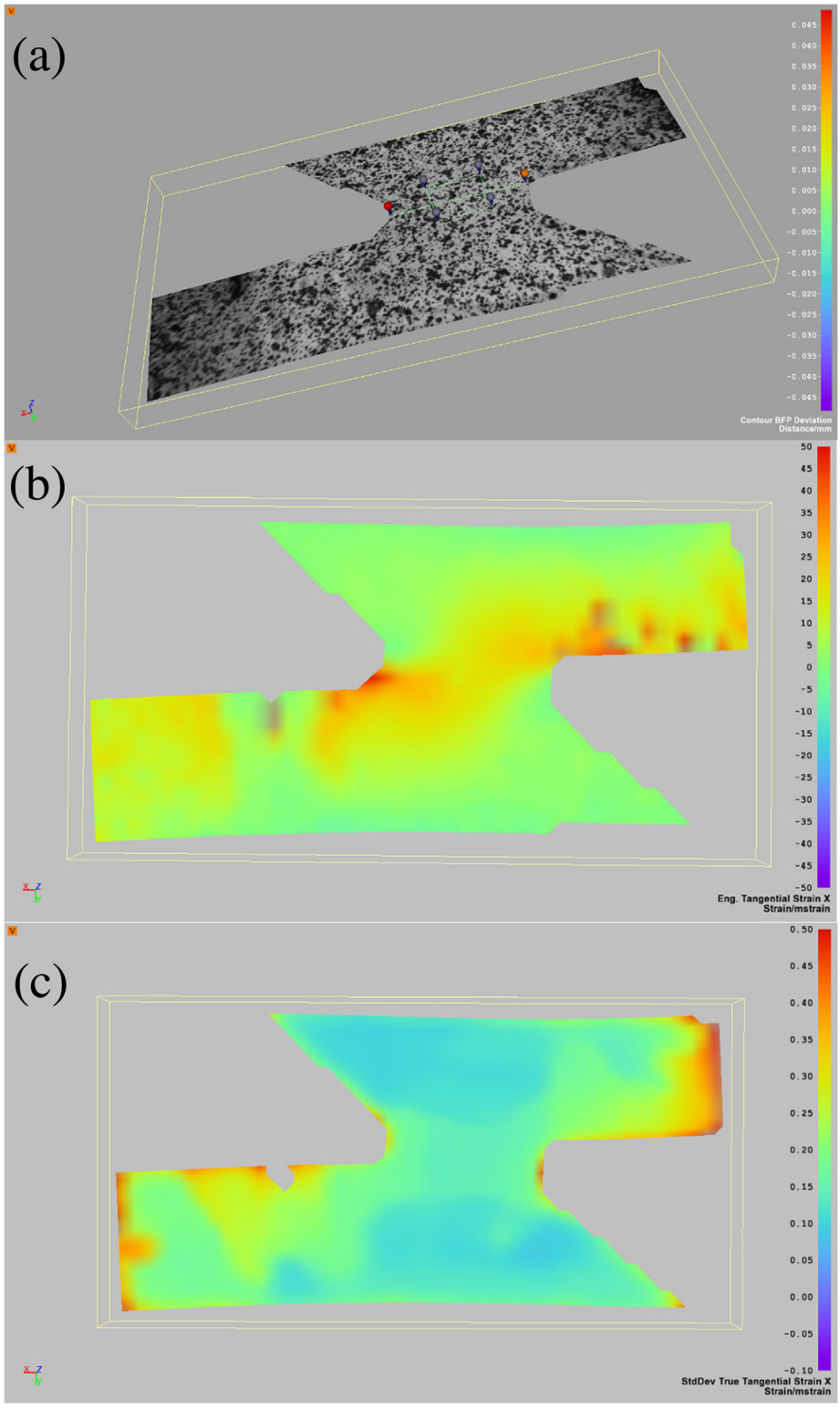

Fig. 7. (a) 3D contour view of a sample tested in a shear test with marked areas, lines, and measurement points. (b) Distribution of ε_{xx} computed in a 3D stereomicroscope DIC system for sample prepared for shear tests (displacement of clamping jaws is 0.485 mm). (c) Distribution of confidence interval of ε_{xx} computed by 3D stereomicroscope DIC software for a sample prepared for shear tests (displacement of clamping jaws is 0.485 mm).

During heat loading of the second sample, photos were taken every degree. The highest temperature was maintained for 15 min, after which time the last photo was taken. Figure 6a indicates the measurement area, lines, and points for which displacements and strains were calculated by the 3D stereomicroscope DIC software. The results of DIC considered ε_{xx}, ε_{yy}, and ε_{xy} are components of the strain tensor in the present study.[8] The distribution of ε_{xx} of the sample treated at 38 °C, and after maintaining it for 15 min at this temperature, is shown in Figure 6b. Based on the analysis of computed results, it can be concluded that as a result of thermal load the sample deforms uniformly in every direction (compare selected results shown in Table 1). The results of measured strains and displacements have low confidence intervals (Figure 6c and Table 1). The temperature of the heating stage, surface of the specimen, as well as ambient temperature was measured by two infrared cameras and by thermocouples. Thus, according to the authors of this paper, usage of micro DIC enables the determination of the effect of temperature change from room temperature to body temperature on the deformation of materials such as biocompatible thermoplastic elastomers with a measurement accuracy of displacements in a sub-micrometer range.

For comparison, the results obtained from the standard DIC system (Q-400 Dantec Dynamics GmbH) by heating the material of a blood chamber up to 38 °C and loading the inner surface of the blood chamber of a VAD using exploitation pressure are $\varepsilon_{xx} = 0.0158$ and $\varepsilon_{yy} = 0.0127$.[17] The components of the strain tensor obtained in the last column of Table 1 using the 3D stereomicroscope DIC system are much lower than the strains obtained previously by the authors,[17] since in this paper the material of the blood chamber was subjected only to a temperature which does not cause such a high strain in the material of the blood chamber as would occur in the case of operating pressure. Thus, the test performed using the 3D stereomicroscope DIC system determines the influence of temperature on the deformation of the material of the blood chamber of a VAD.

The contour view of the last tested sample after processing by the 3D stereomicroscope DIC software with marked measurement points and measurement line is shown in Figure 7a. The ε_{xx} for tensile displacement of 0.485 mm in the shear test is presented in Figure 7b. Also in this case, as in preceding samples made of biocompatible materials of the blood chamber of the ventricular assist device, the results were subjected to a low error, as shown in Figure 7c and in Table 2.

In the FEM simulation of the shear test (based on the in situ scanning electron microscopy micro-shear test) in the central part of the model of the sample for displacement of jaws equal to 0.125 mm, the following were computed: $\varepsilon_{xy} = 0.003$ and $\varepsilon_i = 0.004$.[30,31] In the shear test performed with the 3D stereomicroscope DIC system, in the central part of the sample for the same displacement of jaws, the following were calculated: $\varepsilon_{xx} = 0.00284$, $\varepsilon_{yy} = -0.00187$, and $\varepsilon_{xy} = 0.0014$ (see Table 2). The quoted numbers are averaged values of the central part of the sample. In conclusion, the strains obtained by the 3D stereomicroscope DIC system for the micro-sized samples and simulated in the corresponding FEM model of the shear test loaded by the same displacement have values with a measurement error of a few percent. Thus, the 3D stereomicroscope DIC research method can be used for micro-tension and micro-shear tests for this type of materials.

Table 2. Averaged components of the strain tensor in mm m^{-1} and their accuracy in mm m^{-1} for a contour of a sample examined in a shear test and loaded by selected displacements.

Displacement		0.47 mm	0.125 mm	0.485 mm
Contour	ε_{xx}	1.85 ± 0.12	2.84 ± 0.12	11.45 ± 0.12
	ε_{yy}	-1.22 ± 0.17	-1.87 ± 0.17	-7.34 ± 0.17
	ε_{xy}	0.9 ± 0.2	1.4 ± 0.2	6.7 ± 0.3

4. Conclusions

Conclusions resulting from the presentation of the potential of the newly developed 3D DIC system are as follows:

1) Measurement of displacement and strain on a micro scale with a low error.
2) Measurement of deformation on curved and spherical surfaces.
3) Measurement of deformation in micromechanical tests induced by stretching and/or heating.

Conclusions resulting from the measurement of the deformation of the multi-layer material of the blood chamber of VAD are as follows:

1) Measurement of deformation of material of a blood chamber induced by blood temperature.
2) Measurement of deflection of thin foil induced by the deposition of coating.
3) Measurement of distribution of strain on a surface of a sample tested in a micromechanical shear test.
4) Verification and comparison of data reached in a profilometer test, a microshear test, and FEM models of a shear test.
5) Low measurement errors in all 3D DIC experiments.

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[1] W. H. Peters, W. F. Ranson, *Opt. Eng.* **1982**, *21*, 213.
[2] T. C. Chu, W. F. Ranson, M. A. Sutton, W. H. Peters, *Exp. Mech.* **1985**, *25*, 232.
[3] D. Zhang, D. D. Arola, *J. Biomed. Opt.* **2004**, *9*, 691.
[4] M. Wang, H. Wang, Y. Cen, *Opt. Lett.* **2009**, *34*, 1955.
[5] A. A. Mudassar, S. Butt, *Appl. Opt.* **2014**, *53*, 960.

[6] M. A. Sutton, S. R. McNeil, J. D. Helm, Y. J. Chao, in *Photomechanics*, (Ed: P. K. Rastogi), Springer Verlag, Berlin, Germany **2000**, *77*, 323.
[7] H. W. Schreier, D. Garcia, M. A. Sutton, *Exp. Mech*. **2004**, *44*, 278.
[8] R. Burguete, E. Hack, E. Patterson, T. Siebert, M. Whelan, in *Guidelines for the Calibration and Evaluation of Optical Systems for Strain Measurement*, Michigan State University, Michigan, USA **2010**.
[9] R. Burguete, E. Patterson, *EPJ Web Conf*. **2010**, *6*, 46001.
[10] B. Pan, Q. Wang, *Opt. Exp*. **2013**, *21*, 25056.
[11] M. A. Sutton, X. Ke, S. M. Lessner, M. Goldbach, M. Yost, F. Zhao, H. W. Schreier, *J. Biomed. Mater. Res. A* **2008**, *84*, 178.
[12] B. J. Murienne, T. D. Nguyen, *Opt. Laser Eng*. **2016**, *77*, 92.
[13] Z. Hu, H. Luo, Y. Du, H. Lu, *Opt. Exp*. **2013**, *21*, 11808.
[14] F. Chen, X. Chen, X. Xieb, X. Feng, L. Yang, *Opt. Laser Eng*. **2013**, *51*, 1044.
[15] X. Shao, X. Dai, Z. Chen, X. He, *Appl. Opt*. **2016**, *55*, 696.
[16] S. M. Wilson, A. Bacic, *Nat. Protoc*. **2012**, *7*, 1716.
[17] M. Kopernik, A. Milenin, M. Gawlikowski, I. Altyntsev, R. Kustosz, S. Kąc, *Acta Bioeng. Biomech*. **2015**, *17*, 49.
[18] M. Kopernik, *Adv. Eng. Mater*. **2015**, *17*, 278.
[19] M. Kopernik, S. Kąc, M. Gawlikowski, G. Cios, *Adv. Eng. Mater*. **2016**, *18*, 795.
[20] H. Schreier, J.-J. Orteu, M. A. Sutton, *Image Correlation for Shape, Motion and Deformation Measurements*, Springer, USA **2009**, p. 322.
[21] T. Siebert, T. Becker, K. Spiltthof, I. Neumann, R. Krupka, *Appl. Mech. Mater*. **2007**, *7–8*, 265.
[22] C. Li, Z. Liu, H. Xie, D. Wu, *Opt. Exp*. **2013**, *21*, 15734.
[23] Z. Liu, X. Huang, H. Xie, *Opt. Laser Eng*. **2013**, *51*, 167.
[24] D. I. Kim, Y.-H. Huh, D. J. Kim, C.-D. Kee, *J. Mater. Process. Technol*. **2007**, *187–188*, 232.
[25] X. Huang, Z. Liu, H. Xie, *Acta Mech. Solida Sin*. **2013**, *26*, 570.
[26] M. Gonsior, A. Kapis, M. Darłak, R. Kustosz, P. Jurkowski, Z. Narojek, in *Polskie protezy serca, opracowanie konstrukcji, badania kwalifikacyjne, przedkliniczne i kliniczne*, (Eds: R. Kustosz, M. Gonsior, A. Jarosz), Epigraf, Zabrze, Poland **2013**, p. 582 (in Polish).
[27] J. Kusiński, S. Kąc, in *Technologie w Inżynierii materiałowej i technologie metrologiczne dla potrzeb polskich protez serca*, (Eds: R. Kustosz, M. Gonsior, A. Jarosz), Epigraf, Zabrze, Poland **2012**, p. 120 (in Polish).
[28] S. Timoshenko, *J. Opt. Soc. Am. Rev. Sci. Instr*. **1925**, *11*, 233.
[29] S. Timoshenko, *Strength of Materials*, D. Van Norstrand Company, Lancaster, USA **1940**, p. 360.
[30] D. Saladra, M. Kopernik, *J. Microsc*. **2016**, *265*, DOI: 10.1111/jmi.12431.
[31] M. Kopernik, A. Milenin, S. Kąc, *Arch. Metal. Mater*. **2015**, *60*, 795.

