

Estimating the Porosity of Nickel-Titanium (NiTi) Implants Using Optical Coherence Tomography and Machine Learning

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Abstract. Nickel-titanium (NiTi) implants take root well in the body and have high biocompatibility with respect to surrounding tissues. Porosity is an important parameter responsible for the biocompatibility of the material, and as a result, it is important to be able to control the material manufacturing process to achieve the required values. Destructive and non-destructive methods are currently used to analyse porosity, of which non-destructive ones are the most preferable. A method of estimating the porosity of NiTi materials using optical coherence tomography and machine learning was developed. The trained support vector machine with a radial basis function kernel classifier using the first and the second order statistics of optical coherence tomography images as a feature vector provided the classification accuracy of $95 \pm 6\%$ for two classes of NiTi materials. © 2024 Journal of Biomedical Photonics & Engineering.

Keywords: NiTi implants; optical coherence tomography; machine learning biocompatibility; porosity.

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1 Introduction

Nickel-titanium (NiTi) alloys are widely utilized in medical applications due to their unique properties, such as high biocompatibility, shape memory effects, and superelasticity. These properties make NiTi a suitable material for implants in orthopedics, dentistry, and cardiology, as it can adapt to patient's anatomical features and withstand dynamic physiological loads [1]. The use of NiTi allows for the creation of implants, which are not only match the mechanical properties of natural tissues but also promote tissue regeneration [1].

Biocompatibility refers to how a material interacts with human cells and tissues without causing their significant damage. It depends on factors such as corrosion resistance and the low cytotoxicity [2]. To improve the biological response, materials must have the ability to interact favorably with the biological environment, promoting cell adhesion and proliferation [3].

An essential factor of biocompatibility is the porosity of the implant material [4, 5]. Porosity affects cell and tissue survival by providing space for cell attachment, migration, and vascularization. The strength of implant attachment increases due to bone tissue development inside the pores, while good biocompatibility is ensured by the ability of cells to adhere and spread on the material's surface, forming a monolayer. Surface roughness has a direct beneficial effect on cell morphology and proliferation, whereas a smooth surface hinders cell adhesion, reducing biocompatibility [6].

Porosity is defined as the voids within a material and is characterized by the interconnections, or throats, between these pores, and the walls or struts of the medium that form the three-dimensional structure [7]. Features of pores, throats, or struts are characterized by their size, shape, organization, density, and homogeneity. Traditionally, pore scale is delineated as macroporous, when porous widths are larger than 50 nm, mesoporous, when porous widths are between 2 nm and 50 nm, and microporous, when widths are smaller than 2 nm [8].

Pore volume is typically calculated using the density of the material (ρ_m) and the mass of the sample (m_s) [9, 10]. Eq. (1) shows these calculations of percent porosity, where V_p is the pores' volume, V_T is the total bulk volume of the material and (ε) as either a percent or ratio of V_p to V_T [5]:

$$\% \text{ Porosity} = 100 \cdot \varepsilon = 100 \cdot \frac{V_p}{V_T} = 100 \cdot (V_T - \frac{m_s}{\rho_m})/V_T, (1)$$

Methods allowing to measure or evaluate pores are divided on destructive and non-destructive ones [11]. Destructive methods include hydraulic, sorption, electromagnetic and ionizing techniques. For example, non-destructive methods include pneumatic and visual-optical porosity one. Using pneumatic methods, it is possible to determine the porosity, size and volume of open pores, and the specific surface area of the pores. However, they have limited applications, making visual-optical methods of pore assessment more appealing (see Table 1).

Scanning Electron Microscopy (SEM) is an electron-optical method widely used for observing and examining images of material surfaces, including medical implants, to assess their compatibility with tissues and cells. In study [12], pore size distribution was investigated using SEM images. The researchers utilized SEM images of activated carbon derived from coconut shell charcoal, with an image resolution of 1280×960 px. These images were processed using a program, which applies a threshold value to define areas identified as pores. This

analytical approach can also be adapted to determine the pore characteristics of biomedical materials. SEM allows for the acquisition of high-quality images of porous NiTi, providing clear visualization of the structure and surface of implants produced by various methods, such as high-temperature synthesis processes and capsule-free hot isostatic pressing with argon expansion [13]. These detailed images enable a thorough analysis of pore size, distribution, and interconnectedness, which are crucial factors in assessing the biocompatibility and mechanical properties of NiTi implants.

Optical microscopy is widely used for analyzing porous structures and scaffolds, because it allows for a qualitative assessment of morphology, pore size, and pore interconnectivity. One of the advantages of this technique is its simplicity, as well as the minimal level of sample preparation required [10]. However, optical microscopy has the following limitations: it has a limited depth of focus, making it challenging to obtain high-quality and sharp images at different depths. While it allows for the visualization of pores in their entirety, evaluating porosity often requires knowledge of the depth at which these pores are located, which is not always possible with optical microscopy.

Optical Coherence Tomography (OCT) emerges as an alternative method due to its rapid measurement capabilities, minimal sample preparation, and ability to provide high-quality three-dimensional images of internal structures [14]. OCT allows for horizontal and vertical projections of the sample, giving more complete information about the diameter and size of pores.

Table 1 Comparison of methods for porosity assessment.

Technique	Advantages	Disadvantages	Ref.
Scanning Electron Microscopy (SEM)	Non-destructive; minimal preparation actions; wide-array of applications; the detailed three-dimensional and topographical imaging; easy to operate; visual estimation of interconnectivity, cross-section area, and anisotropy of the pores; greater depth of field, compared to optical microscopy.	Limited depth of focus; concerns in focusing materials with low opacity (i.e., polymers); edge effects due to scaffold preparation; the preparation of samples can result in artifacts; risk of radiation exposure; big size and high cost; black and white images.	[10, 15]
Optical microscopy	Non-destructive; low sample preparation; does not require much time; simply and not expensive.	Low opacity of most of the materials used for scaffold fabrication; difficulties in focusing 3D porous structures; smaller depth of field, compared to SEM; light reflections can mask certain features; color differentiation available; cannot go beyond around 200 nm resolution laterally and 600 to 700 nm axially.	[10]
Optical Coherence Tomography (OCT)	Non-destructive; low sample preparation; does not require much time; simply and not expensive; can produce in real time a two-dimensional image in the space (lateral coordinate, axial coordinate); real-time visualization.	Media opacities can interfere with optimal imaging.	[14]

Table 2 Chemical composition, average particle size, bulk density for powders PTM-1, PTOM-2 and PNK-OT4.

Powder	Chemical composition (impurities, wt. %)	Average particle size, μm	Bulk density, g/cm^3
PTM-1 (Ti)	Ti – Bal.*, N – 0.08%, C – 0.05%, H – 0.35%, Fe/Ni – 0.40%, Si – 0.10%, Cl – 0.004%	45	1.02
PTOM-2 (Ti)	Ti – Bal.*, N – 0.20%, C – 0.05%, H – 0.40%, Fe/Ni – 0.40%, Si – 1.00%, Cl – 0.004%	45	1.36
PNK-OT4 (Ni)	Ni – Bal*, Ni – 99.9%, C – 0.15%, Fe – 0.0015%, Co/Zn/Cu — 0.001%, Cd/Sn/Sb – 0.0003%, Mn – 0.0005%, Pb – 0.0002%	12	1.66

* Balance (Bal.) refers to the main element that constitutes the remainder of the material after accounting for all other specified elements, it fills up the remaining percentage of the composition.

Recent publications demonstrate the effectiveness of this method for the analysis of samples porosity [16, 17]. Therefore, the use of OCT in analyzing the porosity of biomedical materials is appropriate and relevant, especially in serial production and quality control of implants.

Given the volume of data obtained during material analysis, automatic pore detection from images becomes increasingly important. Methods of mathematical morphology and image processing allow for the identification of pores and analysis of their characteristics [18]. To increase the accuracy and speed of analysis, machine learning (ML) methods are increasingly used, capable of processing large amounts of data and identifying complex patterns. ML methods, such as neural networks and other artificial intelligence algorithms, can automatically classify structures in images and quantitatively assess porosity with high accuracy, enhancing the efficiency and objectivity of the analysis.

While ML is increasingly employed to evaluate the mechanical properties of materials, its application in porosity analysis remains limited. Therefore, combining OCT with ML provides an advanced approach to effectively assess the porosity of biomedical materials. The aim of this work is to develop a predictive model for estimating the NiTi porosity using OCT and ML.

2 Materials and Methods

2.1 NiTi Preparation

To produce the porous samples, we prepared a powder mixture by combining two titanium powders, grades PTM-1 and PTOM-2, in an equal mass ratio of 1:1. This titanium powder mixture was then mixed with nickel powder of grade PNK-OT4, ensuring that the total mass of the titanium powders was equal to the mass of the nickel powder (resulting in a Ti ratio of 1:1 by weight). The powders were dried in a laboratory vacuum oven Daihan Sci (South Korea) at a temperature of 70 °C and a pressure of 0.1 MPa for 8 h, then mixed in a mixer for 8 h. The resulting powder mixture was filled into a quartz

tube 400 mm long and 27 mm in diameter, which was then placed in the reactor SOUL-0,4.4/12 (Russia). The chemical composition, average particle size, and bulk density of the powders are shown in Table 2.

The next methods use for fabrication of porous NiTi shape memory alloys: element powder sintering, self-propagating high-temperature synthesis (SHS), hot isostatic pressing, capsule-free hot isostatic pressing, spark plasma sintering, metal injection molding, and mechanical alloying [19, 20].

Porous samples of NiTi were obtained using SHS method in two modes: constant and pulsating. The used quartz tubes had a length of 400 mm [21]. In the constant mode, the combustion process and synthesis reaction proceed uniformly and stably. After initiation with a molybdenum spiral at a temperature of 480 °C, the temperature in the reaction zone increased to approximately 1200 °C and is maintained at this level without significant fluctuations in temperature or reaction rate (deviations do not exceed ± 10 °C). The reaction front propagated at a speed of 80 mm/s. This leads to the formation of a porous alloy with a uniform pore structure and stable characteristics.

In the pulsating mode, combustion and synthesis were characterized by periodic fluctuations in temperature and reaction rate. For obtaining a porous sample in the pulsating combustion mode, argon was used, supplied into a steel flow-through tubular reactor under a pressure of 0.1 MPa. After initiation with a molybdenum spiral at a temperature of 250 °C, the temperature in the reaction zone periodically raised to 1300 °C and drops to 900 °C, creating areas with high and low reaction intensities. The reaction front propagated with average speed of 50 mm/s. These fluctuations could cause inhomogeneities in the structure of the porous alloy, leading to variations in pore size and distribution. The SHS process is schematically shown in Fig. 1.

From the obtained cylindrical porous samples, discs with a thickness of 10 mm were cut using an ARTA-123PRO (NPK Delta-Test, Russia) wire-cut electrical discharge machine for subsequent examination by OCT method.

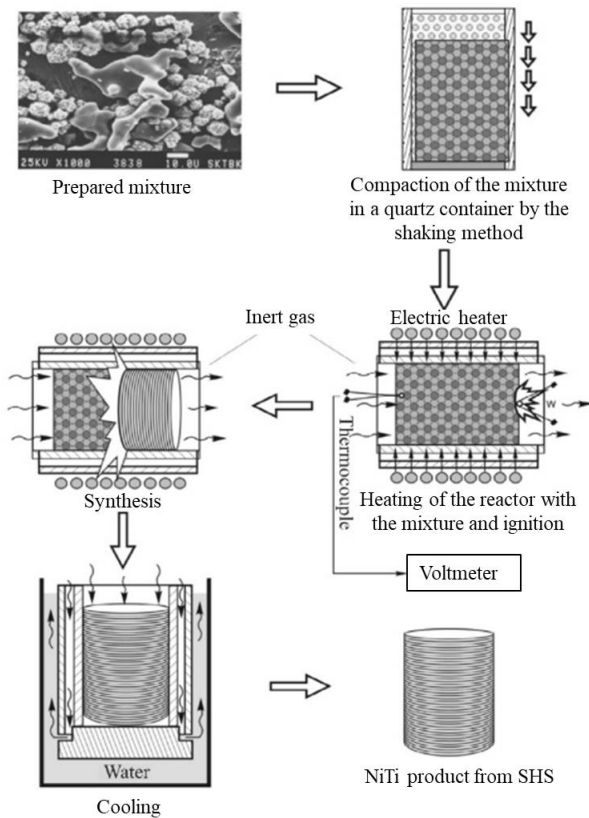


Fig. 1 Scheme for obtaining semi-finished products of porous NiTi in a flow reactor [21].

2.2 OCT Protocol

The study was conducted using OCT on the GANYMEDE-II system (Thorlabs, USA) with the basic scanning module OCTG-900. A superluminescent diode with an operating wavelength of 930 ± 50 nm, used in the GANYMEDE-II system, allows to achieve a signal penetration depth of up to 2.9 mm with an axial resolution of up to $6.0 \mu\text{m}$. During digital data processing, depths from 200 to $400 \mu\text{m}$ were investigated.

Data processing was carried out using ThorImageOCT 5.0.1., with the following parameters: size $500 \times 500 \times 1024$ px, field of view $3 \times 3 \times 2.94$ mm, and pixel size $6 \times 6 \times 2.9 \mu\text{m}$. The experiment was repeated with new disjoint scanning areas, which were highlighted by parallel translation along X, Y-axes and areas were measured from both sides. A total of 20 samples were measured (10 samples in each group). For each sample, 20 C-scans were measured from different non-overlapping areas (400-scans in total were measured).

Statistical analysis and data processing were carried out in Python 3.10 using libraries (numpy, scipy, matplotlib, scikit-image, scikit-learn, PIL).

3 Results

The 3D images were analyzed manually and the depth on the surface of the material was selected. After selecting

the depth, images were divided on non-overlapping areas of 250×250 px (Fig. 2a, b) and averaging was performed by depth to reduce the noise component (Fig. 2c, d). The 95th quartile was chosen in the averaging procedure. The choice of such a procedure is due to the fact that the sample did not have a flat surface and there is an uneven distribution of intensity across the sample, as shown in Fig. 2 a, b. In addition, this procedure showed itself to be optimal in the further processing. Two examples of image processing of a porous NiTi alloy obtained by sintering, using a constant and pulsating mode, before and after averaging are shown in Fig. 2. In total, 1600 images were obtained (see the examples in Fig 3.).

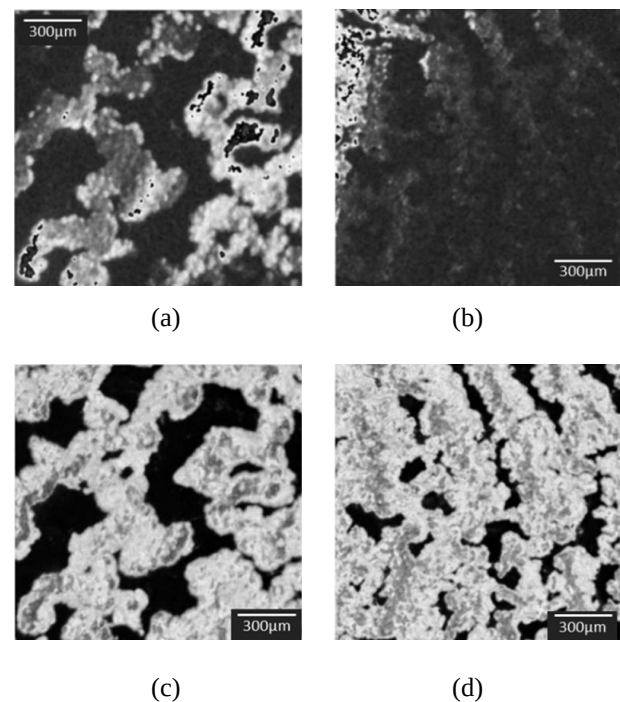


Fig. 2 Examples of images of a porous NiTi alloy obtained by sintering, using (a) a constant and (b) pulsating mode, (c) before averaging and (d) after, respectively.

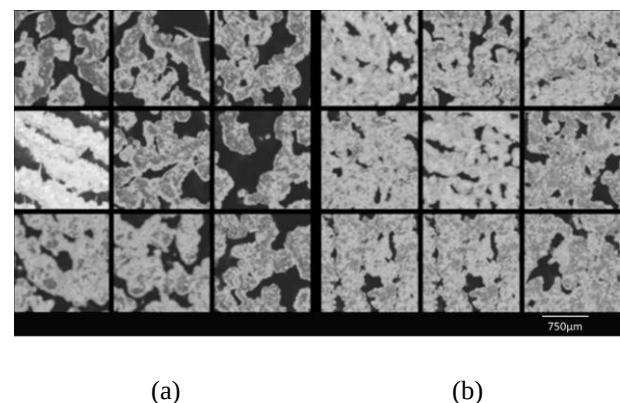


Fig. 3 Examples of images of a porous NiTi alloy obtained by sintering, (a) using argon and (b) using pulsating mode: OCT data were from the depth of $200 \mu\text{m}$, image size was 1.5×1.5 mm.

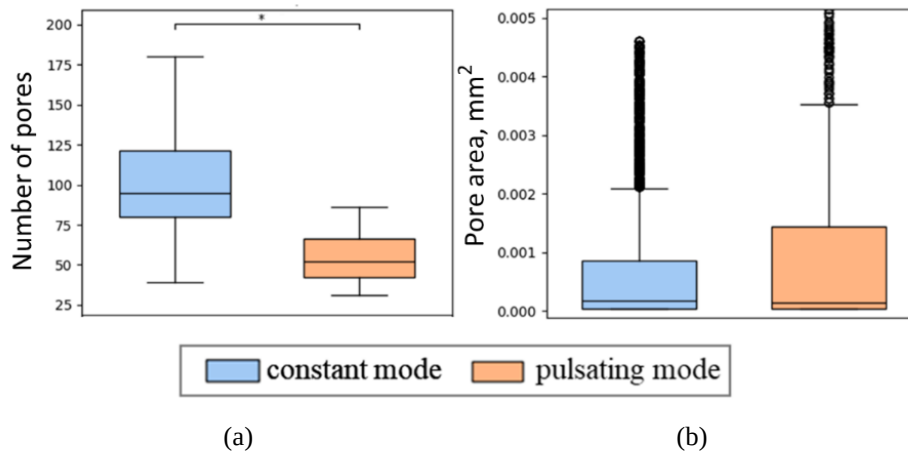


Fig. 4 Distribution of samples (a) by the number of pores and (b) by the pore size. * $p < 0.01$ significance level between group by Mann-Whitney U-test.

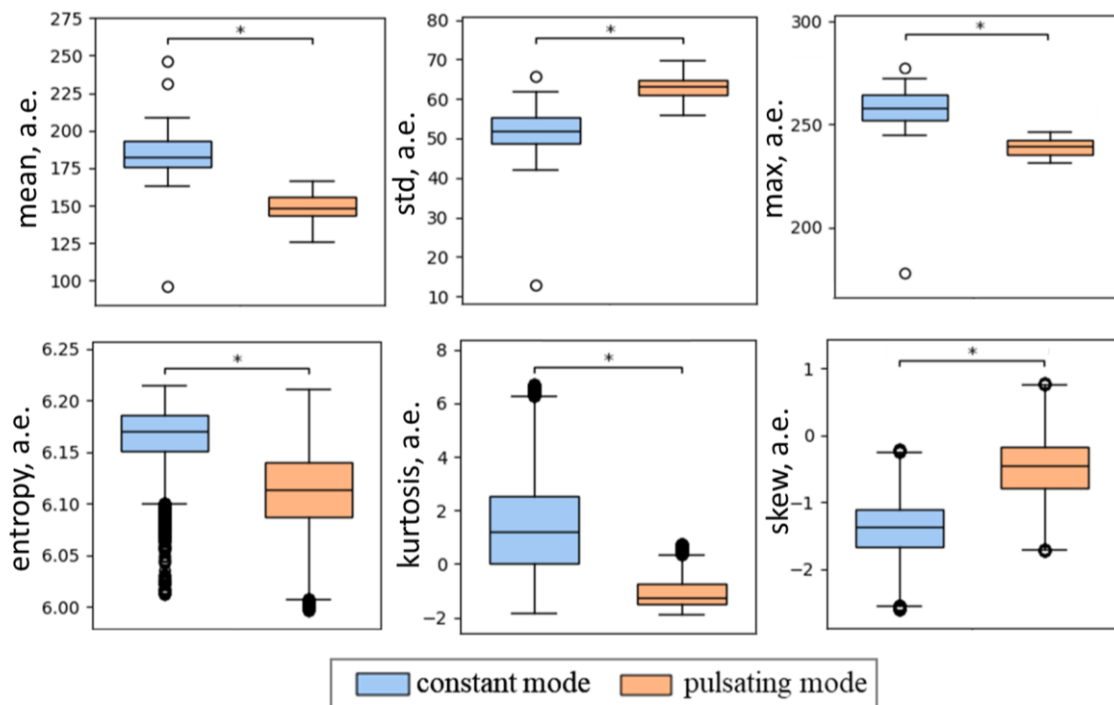


Fig. 5 Distribution of the first order statistics of OCT images. * $p < 0.01$ significance level between group by Mann-Whitney U-test

The next step was to calculate the number of pores and their sizes. The pore segmentation was done using the Otsu threshold, the number of regions was calculated using mathematical morphology, and the search for connected components. The pore area parameter did not show statistically significant differences in the samples, while the number of pores for the sample in the constant sintering mode category differed from the pulsating mode (see Fig. 4).

Despite their simplicity, the first- and the second-order statistics have proven to be useful for analyzing structural features in an image [22]. For the obtained data, the first- and the second-order statistics were calculated. From the first order statistics, the following

characteristics were the most distinctive: mean, standard deviation, max, entropy, kurtosis and skew. For the second order statistics, the following parameters were considered: mean, standard deviation, max, Angular Second Moment (ASM), dissimilarity, entropy, energy, homogeneity, contrast. Normality was tested using the D'Agostino and Pearson tests, which verify the null hypothesis that the sample comes from a normal distribution [23, 24]. All resulting distributions were not normal. The statistical significance of differences was tested using the nonparametric Mann-Whitney test with a significance level of 0.01. All presented distributions had statistically significant differences (see box plots in Fig. 5 and Fig. 6).

The next step was to build a classifier capable of splitting the data into two classes. Support vector machine (SVM) with a linear kernel was used at this stage of analysis. The used dataset consisted of 1600 images (800 images from each class). The 5-fold cross-validation was used. According to this approach,

the dataset was divided into 4 strips for training and 1 strip for testing in 80%/20% proportion. After that SVM classifier was trained and tested using this test strip. This procedure was repeated 5 times to use every strip at the test stage ones. After that, results of classifier testing were averaged.

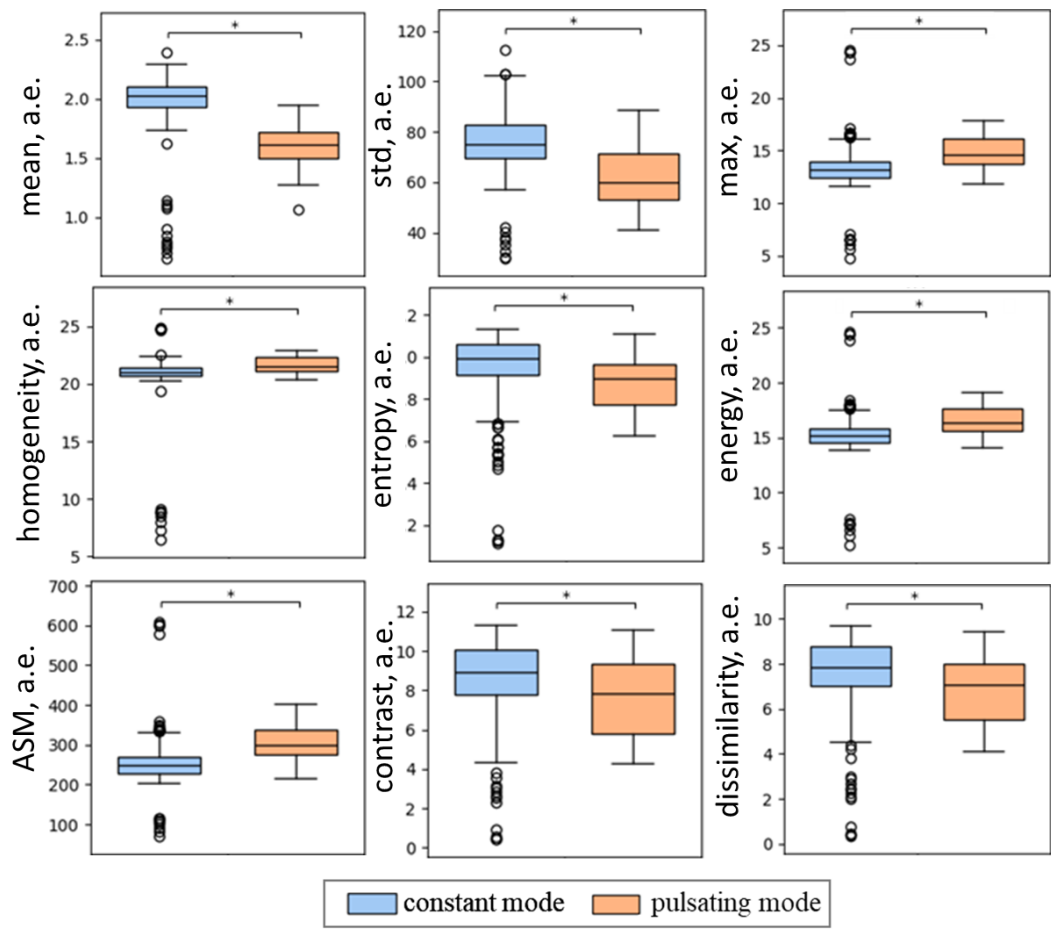


Fig. 6 Distribution of the second order statistics of OCT images.*p < 0.01 significance level between group by Mann-Whitney U-test.

Table 3 Accuracy of SVM classifier with a linear kernel when using the individual first order statistics.

	Kernel	Mean	Max	Std	Entropy	Kurtosis	Skew
Accuracy, % (mean ± std)	Linear	87 ± 8	61 ± 11	76 ± 7	66 ± 12	64 ± 8	61 ± 5
	*RBF	90 ± 5	63 ± 11	78 ± 8	68 ± 13	65 ± 9	64 ± 7

*RBF – Radial Basis Function.

Table 4 Accuracy of SVM classifier with a linear kernel when using the individual second order statistics.

	Kernel	Mean	Max	Std	ASM	Dissimilarity	Contrast	Energy	Entropy	Homogeneity
Accuracy, % (mean ± std)	Linear	89 ± 7	66 ± 11	77 ± 9	66 ± 12	64 ± 8	62 ± 11	66 ± 14	69 ± 14	61 ± 11
	*RBF	92 ± 4	67 ± 12	81 ± 13	68 ± 13	65 ± 9	63 ± 12	66 ± 13	70 ± 10	64 ± 10

*RBF – Radial Basis Function.

Table 5 Accuracy of SVM classifier with Radial Basis Function (RBF) kernel when using the entire group of the first-, the entire group of second-, and the combination of all statistics.

	Kernel	The entire group of the first order statistics	The entire group of the second order statistics	The combination of all statistics
Accuracy, % (mean ± std)	Linear	76 ± 17	79 ± 14	93 ± 4
	RBF	78 ± 13	85 ± 11	95 ± 6

The final classification accuracy for features in the term of individual statistics is shown in Tables 3 and 4. The classifiers performance was in the range of 61 to 92%. The next step was to combine the first and the second order statistics to achieve higher performance. The feature vector for the first-order statistics included 6 parameters, and for the second ones – 9 parameters. Combining all statistics into one feature vector yielded a final feature vector with 15 parameters. The performance of SVM classifier with linear and Radial Basis Function (RBF) kernels on test datasets is presented in Table 5.

4 Discussion

Data models for prediction of the optimal laser parameters in the additive manufacturing (AM) of NiTi shape memory alloys are intensively developed. Two multi-layer perceptron models were created to generate a nonlinear map between inputs and outputs of the AM process [25]. The coefficients of determination (R^2) are around 97–99% for the recovery ratio and transformation temperature. Artificial neural network with multilayer back propagation (BP) learning algorithm was used to predict the mechanical properties of porous NiTi shape memory alloys fabricated by press/sintering of the mixed powders [26]. In comparison with the regression models, the BP model was more effective and can be generally used in conventional press sinter investigation. A model for predicting the porosity of porous NiTi alloy based on SVM was developed [27].

The comparison of prediction values between support vector regression (SVR) and ANN-BP was conducted [28]. The mean absolute percentage error (MAPE) by SVR and ANN-BP were 0.075 and 0.41, respectively. The maximum relative error by SVR and ANN-BP were 10% and 61%, respectively. This shows that SVR has higher prediction accuracy than ANN-BP. Gaussian process regression [29], multiple linear regression, random forest [30], convolution neural network [28] were used to predict porosity in materials manufacturing.

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In this work, the data model to predict the porosity of NiTi materials using optical coherence tomography and machine learning was developed. The SVM with a linear and a RBF kernel, and the first- and second-order statistics of OCT images as coordinates of a feature vector were used. Prediction efficiency was estimated using the 5-fold cross-validation scheme. Two classes of NiTi materials distinguishing accuracy was $95 \pm 6\%$ when all the first- and second-order statistics were included in data feature vector.

The next step is to develop multiclass SVM or SVR data model to predict quantitative characteristics of NITI material porosity.

5 Conclusions

The paper proposes an approach for express analysis of NiTi samples using OCT and SVM. This approach allows for rapid analysis of porous materials with high accuracy. Further development of this approach can be associated with the construction of a regression model for predicting the porosity of samples that have not yet been produced using the manufacturing process parameters. Also, the analysis of the first- and second-order statistics of OCT images of porous materials can be used to control their quality, for example, to search defects.

Acknowledgments

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Data Availability Statement

The data presented in this study are available on request from the corresponding author. The data are not publicly available due to privacy.

Disclosures

The authors declare no conflicts of interest.

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