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Analysis of Particle Size in Composite Materials Using Image Processing

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ABSTRACT

Composite materials are the most important materials in materials science and engineering, which contain two or more materials. In materials engineering, the scanning electron microscopy (SEM) technique is an approach to measure the material's particle size. A new procedure was used instead of SEM is called Artificial Intelligence (AI). Artificial Intelligence (AI) is an interdisciplinary science and branch of computer science that involves solving problems that require human intelligence and capabilities. The computer vision is a subfield of AI, which uses some algorithms to detect the details of images by using computer called image processing. Detecting the particles and measuring the size of materials scanned by SEM is an essential task that helps to describe their feature, traditionally, the size is calculated manually by adding mesh to an SEM image or by drawing a diagonal line in an arbitrary particle. In this paper, a new model based on Artificial Intelligence (AI) is proposed using computer vision to analyze the size of all particles. This model is used to detect the particle size of additives in composite materials like graphene flakes and measure the size of them depending on the reference size fixed on the scanning electron microscope (SEM). The model was used based on the Open-source Computer Vision (OpenCV) library, utilizing multi-layers of canny edge detection, Sobel filter, Brightness and contrast algorithms, using Python 3. The results have achieved very satisfied indication with a very low process time = 0.2 mili-seconds.

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Graphical Abstract



1. INTRODUCTION

In the field of materials engineering, composite materials are the most important branch. Composite materials are consisting from matrix and additives like ceramic, glass, metal, and graphene (1, 2) These additives or some time called filler can be added in different amount, shape, and size to improve the mechanical and physical properties of the matrix (3-5). The size of these additives can be ranged from micron to nanometers, which is important in the field of nanotechnology. Nanotechnology is the science that concerned with the study and preparation of materials with nanometric scale (10^{-9} meter) (6). Nanomaterials is a part of nanotechnology, which can be included organic and non-organic materials like silver, zinc, carbon nanotube, and graphene (7-9). One of the methods to characterize the nanoparticle size in the field of materials science (10) and engineering is scanning

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electron microscopy (SEM) (11, 12). SEM imaging characterization was used to obtain the morphology of the samples as well as distinguish the particles. SEM used the beam of electrons toward the surface of the sample to generate high-resolution images with some details about the topography, composition, and distribution of the particles in the specimen (13, 14). The increasing demand for accuracy in measuring and analyzing nanoparticles within materials, and because the SEM technique is inaccurate way to measure the particle size and requires time (15, 16). The computer vision is a subfield of Artificial Intelligence (AI) (17, 18), which uses some algorithms to detect the details of images by using a computer called image processing (19). Image processing (20, 21) is the process by which one can obtain useful information about any image. Image processing was used in engineering application like cyphering (22, 23) chemistry (24, 25), materials (26), biomedical (27-29), and nanotechnology (30-32), Telcomunications (33), Covid19 detection (34). Image processing has become a radical, fast, and accurate solution for measuring the size of nanoparticles (4, 35). In this study, the image processing was used to measure the size of graphene flakes from the SEM images. In image processing technique, various alogarithms was used and compared among all these logarithms. The logarithms were used in this project include Gamma correction, Sobel (Sobel x and Sobel y), Negative, and Canny edge detection.

2. EXPERIMENTAL METHODS

Five famous preprocessing algorithms were applied and investigated to SEM images, which are include Gamma Correction, Sobel X, Sobel Y, Negative, and Canny edge detection.

Preprocessing Algorithms

2.1. Gamma Correction The parameter used in the power-law function that governs the correction of the image is called gamma. Gamma correction was used to adjust the intensity values of an image and to ensure that the perceived brightness in the image is closer to how the human visual system perceives light (36). Gamma correction was used to compensate the non-linear relationship between pixel values and the actual light levels captured by the imaging sensor or displayed on a screen (37). In many imaging systems, the relationship between the pixel values and the actual light intensity is not linear. The non-linearity behavior can present the images too dark or too bright, especially in the mid-tones. Therefore, to correct the non-linearity intensity by gamma correction increase the pixel values to a certain power. If the value is 1, which is mean no correction linear mapping, while if the values less than 1 (typically

between 0.5 and 2.5) indicate a nonlinear correction as shown in the following equation:

$$Y = X^{\gamma} \tag{1}$$

Where:

Y is the output value, X is the input value, which refers to a pixel in the plain image, and γ is the gamma value (36).

2.2. Sobel (Sobel X and Sobel Y) The image intensity was calculated by Sobel operator at each point, so the Sobel operation was searched the maximum and minimum gradient values in the first derivative of the image. These gradients have certain magnitude as well as direction, so the value of gradient of the pixel was then calculated in a black and white image, which is use 2 kernels with 2 directions i.e., x and y. There are also a convolution masks, which is a small matrix applied to pixel value to obtain a new effect like edge detect. The Sobel filter uses two 2 x 2 kernels. One for changes in the horizontal direction G_x and the other for changes in the vertical direction G_y as shown in matrixes 2 and 3 (38, 39).

$$Gx = \begin{bmatrix} -1 & 0 & 1\\ -2 & 0 & 2 \end{bmatrix}$$
(2)

$$Gy = \begin{bmatrix} -1 & -2 & -1 \\ 0 & 0 & 0 \end{bmatrix}$$
(3)

2.3. Negative The brightest areas are converted into the darkest and the darkest areas are converted into the brightest is called negative. The negative process was occurred by subtracting each pixel's intensity value from the maximum intensity value possible for the given pixel format. For example, in an 8-bit grayscale image (with pixel values ranging from 0 to 255) (40), the negative of a pixel value P is calculated as shown in equation below. In case of color images, the negative operation was performing on each color channel independently. The apply negative operation to an image can produce an interesting artistic effect and highlight certain features or details, especially when the original image has strong contrasts or patterns (41).

Negative_P =
$$255 - P$$
 (4)

2. 4. Canny Edge Detection The Canny is an essential algorithm used for multiple level image edge detection, which was proposed for the first once by Canny (42). The canny algorithm purpose was detected edges with very low error rate. The detected edges should be close to real edges by selecting the best local then labeling the concerned edges.

2. 5. Gaussian Blur Gaussian blur operation was applied to smooth and reduce noise in the image. It

was performed by convolving the image with a Gaussian kernel. The formula for a 2D Gaussian function as shown in Equation 5 (43):

$$Gxy = \frac{1}{\sqrt{2\pi\sigma^2}} e^{-\frac{X^2 + Y^2}{2\sigma^2}}$$
(5)

where:

G(x, y) is the Gaussian function at position (x, y),

 π is the mathematical constant pi (approximately 3.14159),

 σ is the standard deviation of the Gaussian distribution, controlling the amount of blur

2. 6. Gradient Calculation The Sobel operator was used to calculate gradient magnitude and orientation, which is computes the derivatives in x and y directions. Equation 6 was used to calculate the gradient magnitude (G) at each pixel and Equation 7 was used to calculate the gradient orientation (θ) at each pixel, so the angle θ indicates the direction of the edge [46]:

$$G = \sqrt{X^2 + Y^2} \tag{6}$$

$$\theta = \arctan\left(\mathrm{Gy}\,/\,\mathrm{Gx}\right) \tag{7}$$

where: Gx the x-direction gradient Gy the y-direction gradient

3. METHODOLOGY

In this manuscript, a new approach is proposed to calculate the particle size in SEM images by using Image processing. This is done by applying a preprocessing algorithm on the raw image in order to enhance the edges that help particle recognition. Figure 1 shows the process diagram of the work:



Figure 1. The process diagrams

Using an image processing approach to detect objects, these objects may have different shapes, this detection depends on the edges of them, edges detection depends on sharpening the contrast of colors, as denoted in Figure 2.

General procedure was applied to each algorithm as shown algorithm 1 below:

Algorithm 1: General procedure that is applied to each
algorithm used in this work
Step1: Start
Step2: Import important libraries
Step3: Read the input image
Step4: Convert to gray type
Step5: Apply the specific algorithm
Step6: Save the new image
Step7: Stop

The philosophy of object detection and size measurement is to pass a specific mask with dimension (6x6) through over the image, this mask seeks to find edges, and the edges are detected if there is a contrast in color. This contrast is compared with a predefined threshold, in order to detect only edges. Algorithm 2 shows object detection:

Algorithm 2: Object Detection
Step1: Start
Step2: Import important libraries
Step3: Read the input image
Step4: Convert to gray typeStep5: Apply the specific
algorithm
Step5: Pass mask (mxm)
<i>Step6: If the contrast > threshold:</i>
Edges=True
Else
Edges = False
Step7: Draw rectangle for each closed shape
Step8: End
Where $m=6$.

The object measurement is calculated after drawing a rectangle around each object (No matter how the shape looks like!), this is done by treating each rectangle



Figure 2. Different shapes object detection and size calculation

individually, the general idea for calculation and size in the image is shortened by calculating the number of pixels, number of pixels in each object is different from image to other, by referring to specific reference the number of pixels in each unit can be calculated. This can be tuned reaching to the best output.

A Python library like (OpenCV, Numpy, Matplot,) which is a reusable chunk of code were included in this project. After that, read plain image and converted to gray mode to minimize the contrast possibilities. Then each algorithm was applied and saved the new image for the program of size detection.

The object detection was applied after sharpening the edges to measure the size of the graphene flakes. Then using algorithm 3 denotes the procedure of size detection process as shown below:

Algorithm 3: Size Detection
Step1: Start
Step2: Import important libraries
Step3: Read the input image
Step4: Using Gaussian Kernel to remove unnecessary edges
Step5: Find the contours of the image, sort them, and remove
those that are not large enough
Step6: Detect the objects by using contours
Step7: Draw the box and define the Width and Hight (the box
dimensions can be tuned depending on the reference)
Step8: Save the new image
Step9: Stop

The new image which is created by algorithm was read then Gaussian Kernel algorithm was applied to remove unnecessary edges. After that sort contour sorting, which is very valuable in case of image detection or image recognition was applied. In this project two types of sorting contour were used which are included sorting by area and sorting by spatial position. The area sorting was important to extract the large contours representing important parts of an image and get rid of small contours thereby reducing the potential noise. On the other side, sorting by spatial position was also important to sort the characters to the left or right. Finally, drawing box around each graphene flakes and calculate the size from the dimensions (Height and Width), which can be tuned depending on reference object.

3. 1. Results Each SEM image has a scale mentioned in the lower right of image (sometimes in the lower middle). This scale is considered as a reference to calculate the number of pixels in each 100 nm for example, as shown in Figure 3.

This reference is essential for matching the scale between pixels and units, in this work, a manual tuning is done to make matching, this matching is run one time, and then it is fixed for all images. Figure 4 denotes the measurement of the reference after make tuning, in this work the number of pixels in each 100 nm=27.8 pixels. By Applying the proposed model on two SEM images, each has different particles, using correction algorithms to show the effect of each one on the output. Figure 5 denotes the output:

Figure 5 applying the algorithms to the two samples left column sample first and right column sample second:

Tables 1 and 2 show the approximation statistics of graphene flakes size detection from SEM image. The size detection was calculated manually by counting visually the number of particles in the two samples as shown in Figure 5.

Scenario 1: By applying the Canny edge algorithm on the two samples of SEM images as shown in Figure 5 (k, l) the model could detect (58 particles from 66 total particles and 12 particles from 14 total particles respectively) with a high rate of right detection percentage (95% & 100%, respectively), and low wrong detection percentage (5% and 0%, respectively).

Scenario 2: By applying the Negative algorithm in Figure 5 (i, j) shows the high number of graphene flakes particles detected (53 particles from 66 total particles and 13 particles from 14 total, respectively) and high rate of right detection (92% and 100%, respectively) with low wrong detection rate (8% and 0%, respectively).

Scenario 3: By using the Gamma correction algorithm Figure 5 (c, d) shows the number of graphene flakes detection range 47 particles from 66 total particles and 11 particles from 14 total particles, respectively), the right percentage (91% and 100%, respectively) in addition the wrong percentage (99% and 0%).

Scenario 4: The data obtained from Sobel x and Sobel y Figure 5 (e, f, g, h) shows the number of graphene flakes detection range (11 from 66, 3 from 14 for Sobel x and 0 from 66 and 0 from 14 for Sobel y), the percentage of right objects is 0% for all.

In general, the quality of SEM images, and the types of particle shapes may affect the process of detection and even may affect the type of the best algorithm for detection.



Figure 3. Sample of SEM image and highlighting the scale





First Sample

(a)







Second Sample



(b)







Figure 5. Applying the algorithms to the two samples a: Plain image (First sample) b: Plain image (Second sample) c: Particle detection with Gamma edge correction (First sample) d: Particle detection with Gamma edge correction (Second sample) e: Particle detection with Sobelx edge detection (First sample) f: Particle detection with Sobelx edge detection (Second sample) g: Particle detection with Sobely edge detection (First sample) h: Particle detection with Sobely edge detection (Second sample) i: Particle detection with Negative (First sample) j: Particle detection with Negative (Second sample) k: Particle detection with Canny edge detection (First sample) l: Particle detection (Second sample) h: Particle detection (First sample) h: Particle detection (Second sample) h: Particle detection with Negative (Second sample) h: Particle detection with Canny edge detection (Second sample) h: Particle detection (Second sample) h: Parti

TABLE 1. The approximation statistics of graphene makes detection for the first sample								
#All Objects	# Detected	# Right	Right Percentage	# Wrong	Wrong Percentage	Algorithm		
66	47	43	91 %	4	9 %	Gamma		
66	11	0	0 %	11	100 %	Sobel X		
66	4	0	0 %	4	100 %	Sobel Y		
66	53	49	92 %	4	8 %	Negative		
66	58	55	95 %	3	5 %	Canny edge		

TABLE 1. The approximation statistics of graphene flakes detection for the first sample

TABLE 2. The approximation statistics of graphene flakes for the second sample

#All Objects	# Detected	# Right	Right Percentage	# Wrong	Wrong Percentage	Algorithm
14	11	11	100 %	0	0 %	Gamma
14	3	0	0 %	3	100 %	Sobel X
14	0	0	0 %	0	-	Sobel Y
14	13	13	100 %	0	0 %	Negative
14	12	12	100 %	0	0 %	Canny edge

4. CONCLUSION

Detecting the particles and measuring the size of materials scanned by SEM is an essential task that helps to describe their feature, traditionally, the size is calculated manually by adding mesh to an SEM image or by drawing a diagonal line in an arbitrary particle. In this work, image process technique has used, by proposing a new model with different algorithms like Gamma Correction, Sobel X, Sobel Y, Negative, and Canny edge detection. This model has many tasks, first one is to enhance the quality of SEM image by using these algorithms, hence, the detection of each particle depends on particle edges detection, when the contrast of edges is high, then the percentage of detection is high. In general, The results show that the Canny edges and Negative improve the edges rather than Gamma, Sobelx, and Sobely. This result is because of Canny edge detection approach has Gaussian Blur and Gradiant calculation which make this approach good for this work. The effectiveness of each approach deeply depends on the specific characteristics of the image and the requirements of the particular application. Finally, in the future combining multiple edge detection methods or using more advanced techniques, such as the combination of Canny and Sobel, which might yield better results, also propose a new model that surrond the shape as it not as rectangle.

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Persian Abstract

چکیدہ

مواد کامپوزیتی مهم ترین مواد در علم و مهندسی مواد هستند که حاوی دو یا چند ماده هستند. در مهندسی مواد، تکنیک میکروسکوپ الکترونی روبشی (SEM) یکی از راههای اندازه گیری اندازه ذرات ماده است. روش جدیدی به جای SEM استفاده شد که هوش مصنوعی (AI) نام دارد. هوش مصنوعی (AI) یک علم میان رشته ای و شاخه ای از علوم کامپیوتر است که شامل حل مسائلی است که به هوش و توانایی های انسانی نیاز دارند. بینایی کامپیوتر زیرشاخهای از هوش مصنوعی است که به هوش و توانایی های انسانی نیاز دارند. بینایی کامپیوتر زیرشاخهای از هوش مصنوعی است که از الگوریتمهایی برای علوم کامپیوتر است که شامل حل مسائلی است که به هوش و توانایی های انسانی نیاز دارند. بینایی کامپیوتر زیرشاخهای از هوش مصنوعی است که از الگوریتمهایی برای تشخیص جزئیات تصاویر با استفاده از رایانه به نام پردازش تصویر استفاده می کند. تشخیص ذرات و اندازه گیری اندازه مواد اسکن شده توسط SEM یک کار ضروری است که به توصیغ گفتی که می می در است و اندازه می کند، به طور سنتی، اندازه به صورت دستی با افزودن مش به یک تصویر SEM یا کشود یک خط مورب در یک ذره دلخواه محاسبه می شود. در این مقاله مدل جدیدی مبتنی بر هوش مصنوعی (AI) با استفاده از بینایی کامپیوتری برای تجزیه و تحلیل اندازه مواد اسکن شده توسط SEM یک کار ضروری است که به توصیف ویژگی آنها کمک می کند، به طور سنتی، اندازه به صورت دستی با افزودن مش به یک تصویر MEM یا با کشیدن یک خط مورب در یک ذره دلخواه محاسبه می شود. در این مقاله، مدل جدیدی مبتنی بر هوش مصنوعی (AI) با استفاده از بینایی کامپیوتری برای تجزیه و تحلیل اندازه تمام ذرات پیشنهاد شده است. این مدل برای می شود. در این مقاله، مدل جدیدی مبتنی بر هوش مصنوعی (AI) با استفاده از بینایی کامپیوتری برای تجزیه و تحلیل اندازه تمام ذرات پیشنهاد شده است. این مدل برای می شود. در این مقاده می و در می و می در این دانه های گرافن و اندازه گیری اندازه آنها بسته به اندازه مرجع ثابت شده در میکروسکوپ الکترونی روبشی (SEM) می شود. این مرفی در مواد کامپیوتر (OpenCV)، با استفاده از چند لایه تشخیص لیمها، فیلتر Sobel الگوریتمهای روشنایی و کنتر است، استفاده می شود. این مدل بر اساس کتابخانه منبه بر زمانی مرآیند بسیار که ۲.۰ میلی ثانیه دست یافته است.