

Fabrication and wear behavior investigation of the carbon/epoxy composites based on wood using artificial neural networks

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Abstract. Since wood is the main component of the applied raw materials, it can be used as matrix in carbon composites, also it can be taken into consideration as a cost effective advanced application and have this potential to suppress many expensive fabrication and finishing procedures. Wood samples from Oak tree (*Quercus suber*) were heated at different temperatures to produce porous carbon templates. Subsequently, the Carbonized wood was infiltrated with an epoxy in order to fabricate the final carbon/epoxy composite. Scanning electron microscopy was used to elucidate parameters affecting on microstructure and wear properties of products. In this context, artificial neural networks (ANN) and design of experiments method (DOE) was implemented to analyze the wear performance of a new class of cellulose based composites. This work indicates that epoxy shows good reinforcement characteristics as it improves the sliding wear resistance of the carbon matrix and that factors like carbonization temperature, sliding distance and normal load are the important factors affecting the wear behaviors.

Introduction

“In spite of the rapid introduction of wood substitutes, new uses are being constantly found for wood” [1]. Since the time of announcing of that interesting sentence up to now, new uses and applications have been found for this natural outstanding material. In the recent past few years, interests of engineers and researchers have been changed from expensive alloy materials to carbon composite materials. Wood-based carbons and their composites have introduced many novel engineering fields including thermal, mechanical, weight and dimensional sensitivity and also tribological applications. Because of their good dimensional stability and low density compared with metals, alloys, ceramics and even some polymers. In addition, the low cost of wood as a raw material is a remarking point for this category of advanced materials. Carbonized wood achieved from plants in the form of templates, chops and husks can take into account as matrix or reinforcement in composite materials. Using this approach, we are able to keep all anatomical characteristics of wood with a completely different composition [2]. Wood is composed of several natural organic materials which form a complex porous body of different interconnected elongated hollow cells aligned with central axis of the tree trunk. The chemical composition of a sample of dry wood is: 50 wt.% carbon, 44 wt.% oxygen and 6 wt.% hydrogen [3]. Three polymers are the main components of wood: cellulose, hemicellulose and lignin. The substantial structural component in all plant cell walls is cellulose. It is contained in a matrix of the other two polymers with the proportions of the constituents varying depending upon location in the cell wall. Different heat treatment temperatures cause different density of bulk carbonized wood by changing cell wall molecular arrangement in solid carbon [4]. During wood carbonization, at 5 °C/min of heating rate, hemicelluloses is decomposed at temperatures ranging from 170 to 240 °C, cellulose 240–310 °C, and lignin 320–400 °C. Many evolutionary reactions like bonding of wood elements and evaporation of some compounds take place during heating and at last only charcoal left [5]. In the literature, several studies have been performed on many wood

samples carbonization and its different aspects of [6-11]. Fabricated carbon body has too many desired properties like stable coefficient of friction, good electromagnetic shielding, low coefficient of thermal expansion, high damping capacity, self lubricity and excellent far infrared property [12-13]. The main purpose of using reinforcement can be considered to improve the mechanical, thermal or tribological properties and to reduce the cost of the final product as Reinforcements like ceramic or metal is being used in carbon composites to improve the wood-based mechanical and surface properties remarkably [14]. In current research carbon/ epoxy composite was manufactured by infiltrating epoxy polymer into porous carbon body derived from wood. The relation between the material wear properties and the material's own characteristic such as the density of matrix part and the operating conditions is an important point to take care for production and performance of composites in many engineering challenges like tribological applications. Taguchi's design strategy was utilized to concentrate on the effect of various parameters on material wear response. An artificial neural network (ANN) model has been developed to predict specific wear rate with an acceptable computational accuracy. The artificial neural networks which is inspired by the work of human body is very useful and easy tool in different engineering fields. They are so powerful for property evaluations because the problems are usually highly complex and nonlinear. Also, neural network's algorithm has this possibility to learn from a set of examples and generalize the rules to new situations [15].

Experimental

Wood samples from The oak tree (scientific name: *Quercus suber*) was prepared in cubic shape with precise cutting instrument in the size of 20 mm length and width and 10 mm height. Then, the samples were oven-dried for removing their structural water in order to avoid spoiling and fungus disease during tests. As reinforcement, the epoxy resin chemically belonging to the 'epoxide' family and its corresponding hardener are mixed in a specific ratio.

Wood was heated to different temperatures in order to produce porous carbon body as a matrix for carbon/epoxy composite (Fig. 1). Wood pyrolysis process was carried on at temperature ranges between 400 °C to 1100 °C with an interval temperature of 50 °C in a specific argon atmosphere control furnace (10 to 12 samples in each temperature) but only samples which carbonized at 400 °C, 450 °C, 500 °C and 550 °C were used for next wear tests. Epoxy resin was then infiltrated into solid porous carbon body but efore this step, epoxy was preheated for increasing its capability of flowing in carbons porosities. Localized vacuum was set on the bottom of carbon frame to ease and accelerate infiltration process. For each carbonization temperature, density of produced carbon has been recorded and finally, Scanning electron microscopy (SEM) was used to study the microstructure of C/epoxy composite (Fig. 2).

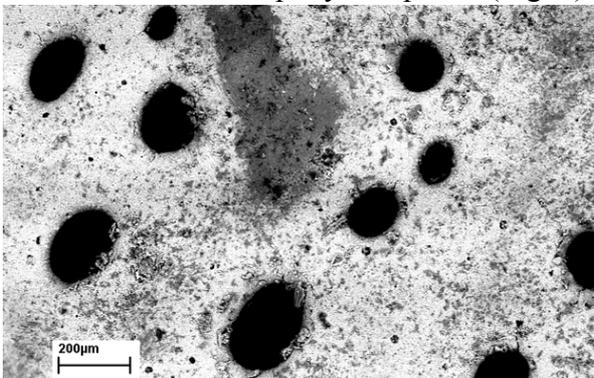


Fig. 1. Microstructure of carbon derived from oak wood

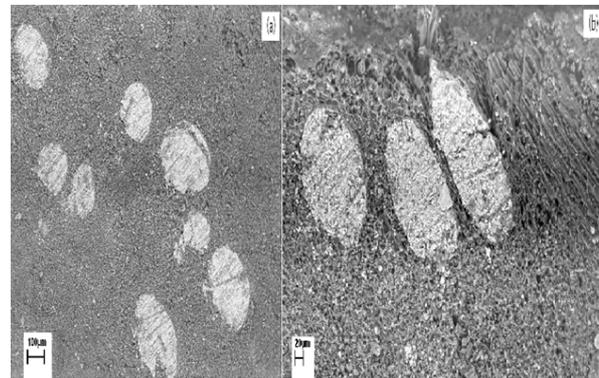


Fig. 2. Microstructure of carbon/epoxy composite in: (a) center and (b) edge part of composite.

The sliding pin-on-disc type wear test was done on the carbon/epoxy composites in air without using any lubricant agent at room temperature. The counter body is made of a hardened steel disc (hardness 72 HRC, surface roughness 0.6 μm Ra). The specimen is fixed and the disc is rotated while a normal force is applied to the upper surface of composites. For obtaining a proper clean and smooth face before tests, both the samples and disc surface were ground with grinding papers. According to Table 1, the wear experiments were carried out using following strategy: four sliding distance of 50, 75, 100 and 125 m, four normal loads of 5, 10, 15 and 20 N and finally, we use composites which their carbon matrix was heated to 400 $^{\circ}\text{C}$, 450 $^{\circ}\text{C}$, 500 $^{\circ}\text{C}$ and 550 $^{\circ}\text{C}$. The reason of choosing these different temperatures will be discussed later. The worn sample mass change in surface is then measured with a sensitive electronic balance with accuracy ± 0.1 mg. The specific wear rate is defined as the volume loss of the specimen per unit sliding distance per unit applied normal load ($\text{mm}^3/\text{N}\cdot\text{m}$) and subsequently, In order to study the worn surface of composite a SEM micrography was conducted. Before taking picture, a platinum thin film was put on the sides of cubic samples for increasing the electrical conductivity of composite materials.

Design of experiment is an analysis tool for analyzing the influence of different test factors on corresponding output. Selection of the control factors is the most significant step in the design. According to full factorial method, $4^3 = 64$ wear tests on the fabricated composite have to be performed under three different operating parameters each at four levels but, by using Taguchi's L_{16} (4^3) orthogonal array only 16 runs were done that put forward a great benefit in experiments time and financial aspects (Table 2). Subsequently, the "smaller is the better" characteristic in the term of signal-to-noise (S/N) ratios was defined for evaluating the minimum specific wear rate according to Eq. 1.

Smaller is the better characteristic:

$$S/N = -10 \log 1/n (\sum y^2) \quad (1)$$

In the above S/N ratio formula n is number of performed experiments and y is the achieved value of data [16]. Table 2 explains design details and corresponding S/N ratios.

Table 1. Parameters and related factors in wear experiment

Control factors	Level 1	Level 2	Level 3	Level 4	Units
A:Carbonization temperature	400	450	500	550	Degree centigrade ($^{\circ}\text{C}$)
B:Normal load	5	10	15	20	Newton (N)
C:Sliding distance	50	75	100	125	Meter (m)

Table 2. Taguchi's L_{16} experiment design and analysis

Test runs	Carbonization temp($^{\circ}\text{C}$)	Normal load(N)	Distance(m)	S/N ratio
1	400	5	50	44.265
2	400	10	75	43.9994
3	400	15	100	42.9504
4	400	20	125	41.8303
5	450	5	75	45.8146
6	450	10	50	45.1141
7	450	15	125	44.265
8	450	20	100	42.8052
9	500	5	100	44.1943
10	500	10	125	43.8358
11	500	15	50	45.7302
12	500	20	75	46.9551
13	550	5	125	44.4225
14	550	10	100	44.027
15	550	15	75	45.7302
16	550	20	50	47.9155

Inspired by human nervous system, the application of artificial intelligence approaches has recently found its place in engineering fields. Therefore, Neural networks, as a branch of artificial intelligence, have used in evaluation of mechanical and physical behavior of composite materials specially when the problem is very complex, non-linear and multi dimensional and in conclusion, when an exact extraction of an analytical solution is very complicated to. Artificial neural networks have the ability of learning directly by examples without any determined relation or constrains. The system was assembled from too many units called neurons or nodes. Neurons linked to each others and transform data from one part to another in order to conduct calculation process. The neural system generally consists of three parts: input layer, hidden layer and output layer. The non-processed data is entered to the input layer. No processing actions take places on this layer. In the hidden layers training and testing jobs happens according to neural networks own algorithms and regulations .It is so called because the results of this layer come to next layer directly and is not accessible for analyzer. The number of neurons in this layer of the neural networks is different depending on problem nature and network architecture. At last the output layer presents information[17].

Results and discussions

Fig. 2 shows the SEM picture of carbon/epoxy composite based on carbonized oak wood. The dried wood first carbonized in an air-isolated condition and then, the porosities of fabricated solid carbon was filled with polymer. While carbonization, the structural shape and conditions of porosities on the wood does not change and this leads to the different separated parts in carbon matrix filled with epoxy in counteract with carbon/polymer particle or polymer/ dust carbon composites. Basically, variation of density and porosity in wood with treatment temperature during carbonization in an isolated condition depends on two factors that they act in opposite direction with each other: (i) Weight loss due to degradation of some wood component while heating and also evaporation of volatile chemicals in wood. This phenomena decrease the density of solid carbon. (ii) Wood cell-wall expansion and shrinkage of wood samples that reduce the pore's diameter and increase density in fabricated product. Approximately, from 400 °C to 1000 °C the second effect is dominant and for higher temperatures the first. The trend of density change with temperature is shown in Fig. 3. In current study we choose temperatures between 400 °C and 550 °C in which density increases with temperature. In practice as temperature increases in the range of study, the compression strength of solid carbon improves and this is because of the densification of internal structure and existence of smaller holes, as stress concentration zones, in the carbon surface. Uniaxial compression test was performed for approval of this effect. Compression strength of porous carbon body at different temperatures is listed in Table. 3.

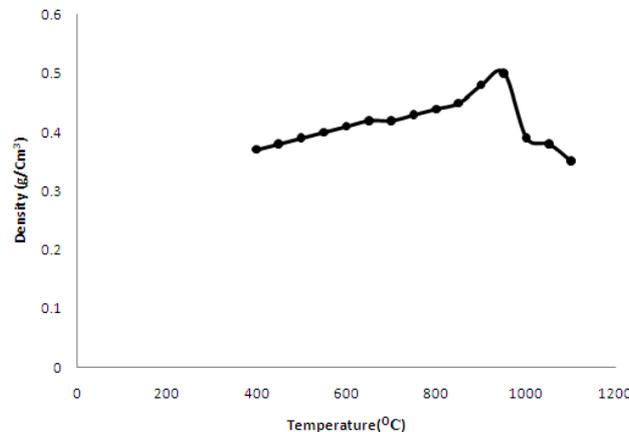


Fig. 3. Density change with carbonization temperature of porous carbon.

Table 3. Mechanical property of porous carbon in different temperatures.

Carbonization temperature(⁰ C)	Compression strength(MPa)
400	29.3
450	32
500	36.4
550	40.1

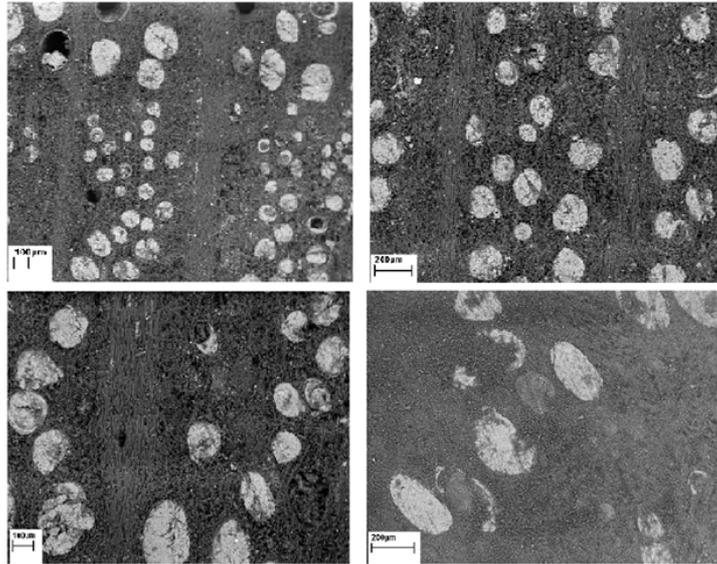
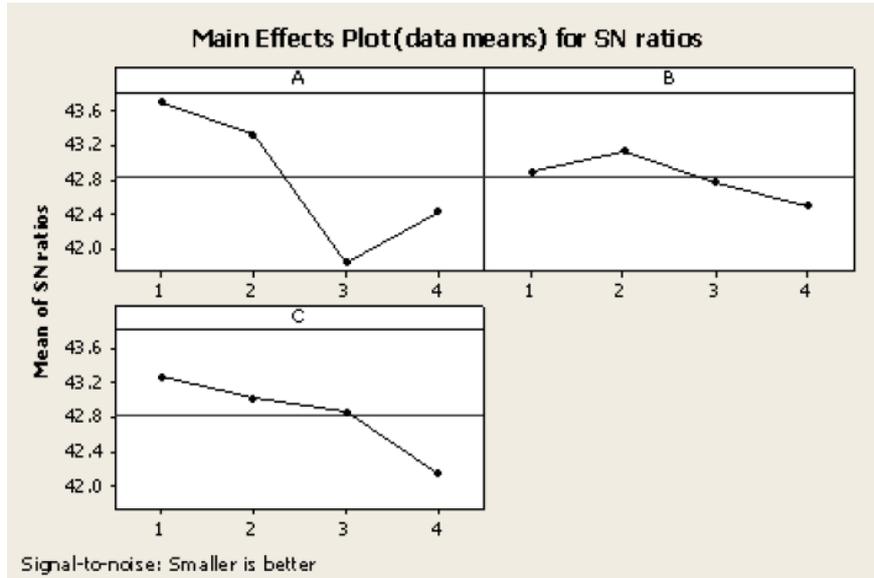
**Fig. 4.** Worn surface of carbon/epoxy composite based on oak wood after test at room temperature.

Fig. 4 illustrates the surface morphology of carbon /epoxy composites after test. In the first rounds of steel disc, some small carbon particles peeled from surface of composite and spread into the whole surface of material by the subsequent moves of counterface. Presence of this carbon particles on the solid carbon body and their lubrication characteristics reduces the specific wear rate of composite ¹³. Carbon fabricated from wood at more elevated treatment temperature has higher strength and peeled harder from composite surface so they take part in fabrication roll much more later so, those samples which their matrix carbonized at lower temperatures shows better enhanced wear resistance. SEM micrograph indicates other important phenomena that are the unbonding between the infiltrated epoxy and the carbon matrix occurred after wear test. Heat generation happens during interaction of disc and material assists pull out by increasing the flow ability of composite (Fig. 4). When polymer pulls out from the carbon porosities, agglomeration take place, lumps of polymer shaped and by adhering on the counterbody reduce the contact of metal disc and composite and wear rate. In contact conditions between polymers and metals, when the metallic surface roughness is greater than 0.05 μm abrasion mechanism becomes the most important factor ¹⁸. So, because of the higher surface roughness of test set up (0.6 μm) abrasion is the main agent of polymer lumps transfer to the disc body.

According to the experiment design and achieved specific wear rate, related S/N ratio for sixteen runs was calculated and is presented in Table 2. The Response table for Signal to Noise Ratios used for “smaller is better” strategy is illustrated in Table 4. Using the prediction ability of taguchi method, the mean of S/N ratio in test is 43.8513. Prediction analysis of wear test shows that the carbonization temperature is the most significant factor among three factors and subsequently, sliding distance is the second and normal load is the third affecting factors of test. The effect of each control factor involved in wear is shown in plotted in Fig. 5. It should be added that the A₁, B₂, C₁ arrange of wear control factor gives the minimum specific wear rate.

Table 4. Response Table for Signal to Noise Ratios

Level	A	B	C
1	43.71	42.9	43.28
2	43.33	43.14	43.02
3	41.85	42.77	42.86
4	42.43	42.51	42.15
Delta	1.86	0.63	1.13
Rank	1	3	2

**Fig. 5.** Effect of control factors on sliding wear rate.

Since the effects of both materials and test operating parameters on specific wear rate is multi-dimensional and non-linear, Wear is taken into consideration as a complex problem which needs a powerful and high accuracy mathematical methodology like soft computation method to be modeled. Artificial neural networks, as one of the most significant categories of artificial intelligence, composed of a number of interconnected neurons, resembling the human brain. A neural network consists of three linked divisions in its structure: input layer, hidden layer and output layer. In order to analyze the composite wear behavior in current study, carbonization temperature, normal load and sliding distance are considered as network input parameters and output layer gets specific wear rate. Each of above input parameters value introduced to model by occupying one input layer neuron. Available data is divided into two sets, one for the neural network training and the other for the testing predicted outputs. Before training, all data were normalized in the range of 0–1 to improve the comparison ability of the relative value of inputs data and at last for representing the actual specific wear rate and calculating accurate errors denormalization was carried on. At constant learning rate, error tolerance, momentum parameter and noise factor in different hidden layer number and architecture, training action was utilized by using a fraction of data sets. A Multilayer perceptrons (MLP) artificial neural network was chosen for specific wear rate prediction. The feed forward back-propagation ANN used here because Back-propagation uses the gradient descent algorithm which has a very good ability in training by examples and extracting logical links between inputs and outputs. The tangent sigmoid transfer function (tansig) was used in the hidden layers, while a linear transfer function (purelin) was used in the output layer. Minimizing the mean square error (MSE) between the predicted outputs and the target is the neural network performance criterion. The four-layer neural network used in this work is shown in Fig. 6.

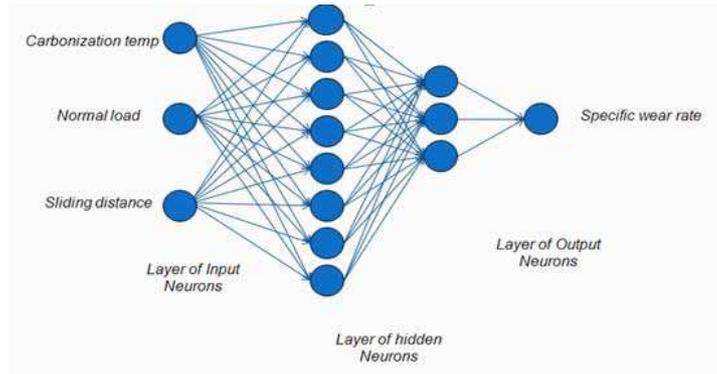


Fig. 6. The four-layer neural networks.

The curves showing the effect of carbonization temperature on specific wear rates of wood-based carbon/epoxy composites at different sliding distances and normal loads are presented in Fig. 7 and 8. Also, the effect of sliding distance at variety of normal loads on specific wear rate is illustrated graphically in Fig. 9.

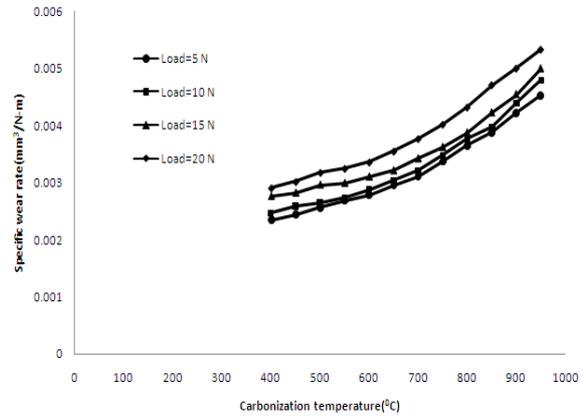
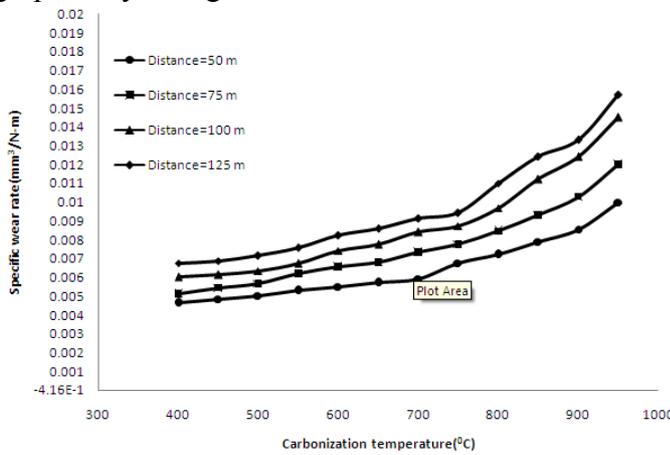


Fig. 7. Prediction of Specific wear rate with carbonization temperature at different sliding distances.

Fig. 8. Prediction of Specific wear rate with carbonization temperature at different normal loads.

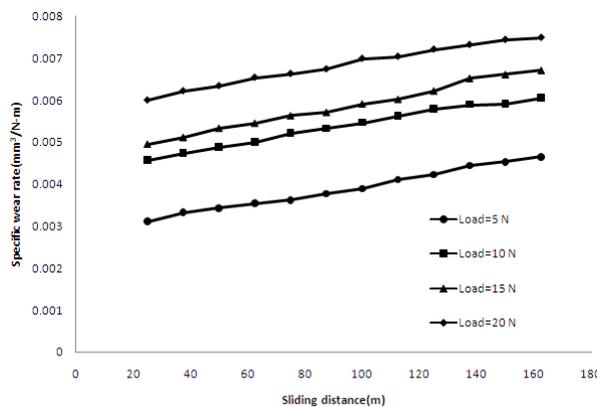


Fig. 9. Prediction of Specific wear rate with sliding distance at different normal loads.

As illustrated in three above figures, it can be seen that the specific wear rate increases almost exponentially with increasing wood carbonization temperature and sliding distance because of the lubricating feature of fine primary eroded carbon and polymer pull out from matrix porosity during completing wear distance. In addition, composite wear resistance is decreasing almost in a linear pattern with increasing sliding distance as one of significant factor of the test.

Conclusion

Dry wood of oak tree with its own intrinsic anatomical porous feather is heated in isolated condition and transforms to solid carbon frame. Because of this outstanding characteristic and natural shape stability of carbon after carbonization, carbon/epoxy composite was produced by infiltration process. Epoxy polymer diffuse into the interminable spherical porosities in carbonized wood and stay separated from other polymer section in the body of composite. Compression test on carbon and wear test on composite was performed. Increasing temperature improve carbon strength also specific wear rates for the final materials increase with increasing the applied load and the treatment temperature. This research indicates that the use of a soft computational methods and design strategy is so effective and helps to recognize the wear resistance of such wood-based carbon composites. The application and efficiency of wood derived materials in new advanced manufacturing processes was represented on this study.

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