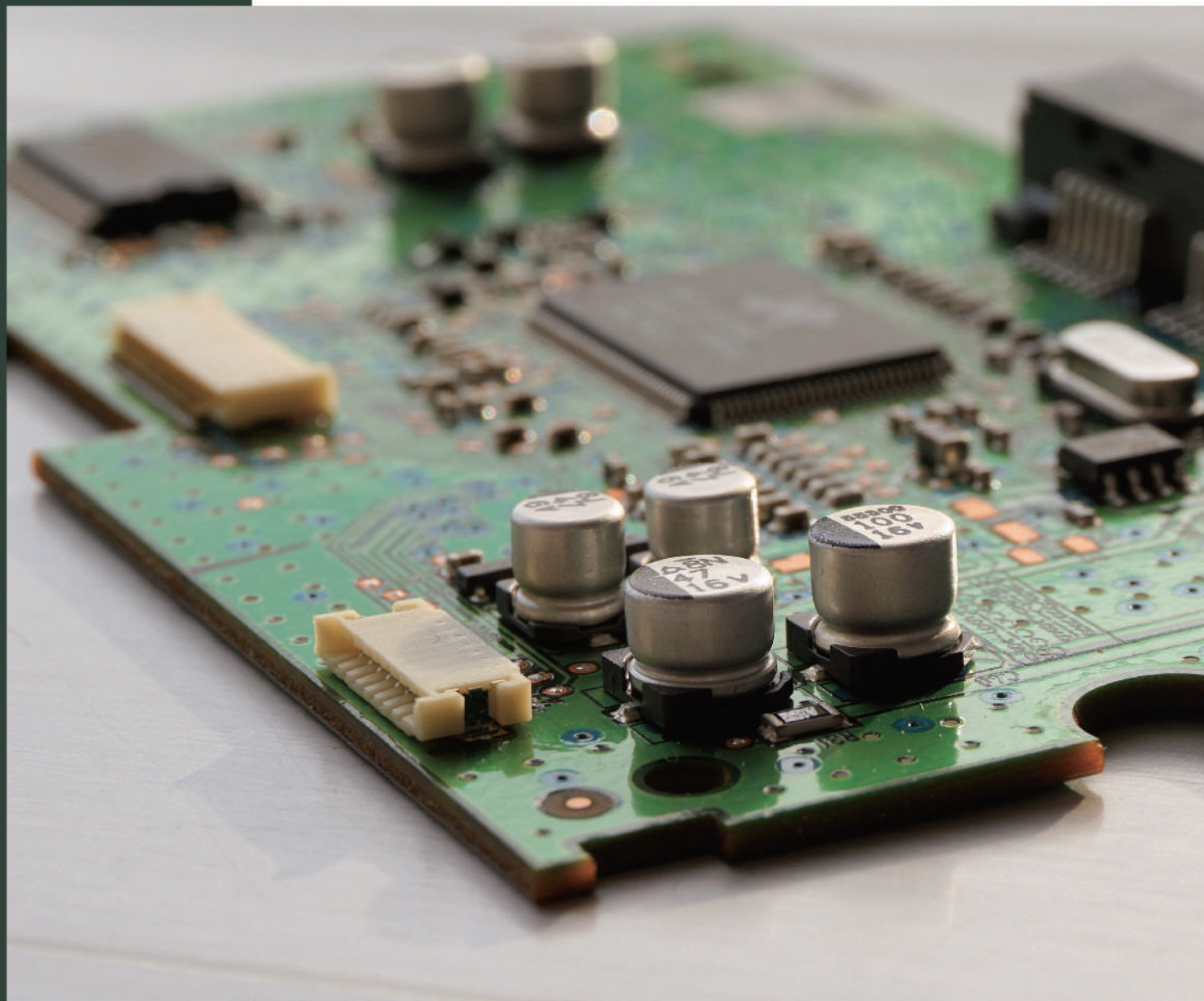


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ARTICLE

Heuristic Order Reduction of NARX-OBF models Applied to Nonlinear System Identification

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ABSTRACT

Nonlinear system identification concerns the determination of the model structure and its parameters. Although the designers often seek the best model for each system, it can be tricky to determine, at the same time, the best structure and the parameters which optimize the model performance. This paper proposes the use of a Genetic Algorithm, GA, and the Levenberg-Marquardt, LM, method to obtain the model parameters, as well as perform the order reduction of the model. In order to validate the proposed methodology, the identification of a magnetic levitator, operating in closed loop, was performed. The class NARX-OBF, Nonlinear Auto Regressive with eXogenous input-Orthonormal Basis Function, was used. The use of OBF functions aims to reduce the number of terms in NARX models. Once the model is found, the order reduction is performed using GA and LM, in a hybrid application, capable of determining the model parameters and reducing the original model order, simultaneously. The results show, considering the inherent trade-of between accuracy and computational effort, the proposed methodology provided an implementation with good mean square error, when compared with the full NARX-OBF model.

1. Introduction

A system can be defined as a structure in which different variables interact and produce observable signals. In the same way, a model expresses the relation between observable quantities. Therefore, allowing the prediction of properties and behavior of an object [1].

In control engineering, modeling systems is a constant need. The model can provide a better understanding of the system operation, it can also be a powerful prediction tool

which may prevent faults in the real system [2].

System Identification can be regarded as the field responsible for relating a purely mathematical model to a system. That is, in the development of the model, only data concerning the system input and output are needed [3]. That approach is known as black box modeling and it is very appealing, since no simplifying assumptions are requested when building the model [4].

There are several classes of models that can be used in the development of a black box model. The designer must consider representation capability and computational ef-

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fort when choosing a class. That is not an easy choice and is often empirically made. As examples of classes of nonlinear models frequently mentioned in literature, one can cite Volterra Series [5] and NARX [6], Nonlinear Auto Regressive with eXogenous input. These classes may present a dimensionality problem, since the number of terms to be determined in the model is often high [3].

The dimensionality problem is less critical in NARX models, since past outputs are considered in the model, which reduces the number of terms required by the model. Additionally, it is possible to reduce the computational cost of NARX models even more, selecting the most relevant series' terms [7] (in other words, performing the order reduction of the model).

Order reduction techniques are specially interesting for the representation of highly nonlinear systems, since the number of terms in a polynomial model increases with the nonlinearity degree of the system [3]. NARX models are known for the high representation capability. In order to avoid the loss of this characteristic, the order reduction must be carefully parametrized [8].

It is worth mentioning that the use of OBF, Orthonormal Basis Functions, is widely known in literature. ARX-OBF [9], which are Infinite Impulse Response, IIR, models and Volterra-OBF [8,10,11], which are Finite Impulse Response, FIR, models have already been proposed. NARX-OBF models [11], which can be seen as a feedback version of the Volterra-OBF models, have also been treated in previous work.

It can be considered that a NARX-OBF model is a more compact implementation of the classical NARX models. That is, the use of OBF reduces the number of terms necessary to the model. In order to improve the performance of the NARX-OBF models, this paper proposes an order reduction methodology for this class of models.

A Genetic Algorithm, GA, is used to select the most representative terms of the full NARX-OBF model. Thus, simplifying the model realization and reducing the simulation time. The evaluation function applied in the GA is inspired by the Akaike Information Criterion [12], AIC. This criterion quantifies the impact in the Mean Square Error (MSE) caused by the insertion of a new term in the model. At the same time, the AIC penalizes the insertion of this new term, since it increases the model complexity.

With a fitness function which considers the AIC, the GA proposed in this work is capable of realize the joint minimization of the MSE and of the number of terms in the model. That is, the GA performs a multi-objective optimization, aiming the simplest model with the best representation.

It is important to mention that the use of GA in the

order reduction of NARX polynomial models has already been investigated [13]. However, in this paper it is proposed the use of GA as a mechanism of search for the poles of the Kautz functions, in NARX-OBF models, and also to reduce the number of terms in the series that implement such a model. Furthermore, in the proposed methodology, the GA acts by interleaving its actions with the Levenberg-Marquardt method, which is the latter responsible for determining the coefficients of the NARX-OBF model.

The main contributions of this work can be defined as:

(1) the joint minimization of: (1) the MSE and; (2) the complexity of the NARX-OBF models, (i.e., the joint search for the model structure and its parameters);

(2) the interleaving application of the GA method (searching for Kautz poles and the model structure), and the Levenberg Marquardt method (which search for models' coefficients).

This paper is divided as follows: section 2 presents the structure of NARX-OBF models, section 3 gives a summarized description of the methods for parameter selection, section 4 presents the identification of the magnetic levitation system, section 5 presents the main results obtained and, finally, section 6 is dedicated to conclusions and future work.

2. NARX-OBF Models

This section is dedicated to present basic concepts regarding orthonormal functions and NARX-OBF models [11].

2.1 Orthonormal Basis Functions

The main property of orthonormal functions is expressed by Eq. (1).

$$\langle \psi_m(k), \psi_n(k) \rangle = \begin{cases} 0 & m \neq n, \\ 1 & m = n, \end{cases} \quad (1)$$

in which $k, m, n \in \mathbb{Z}$, and $\varphi_m(k)$ and $\varphi_n(k)$ are orthonormal functions and $\langle \cdot \rangle$ is an inner product, defined by Eq. (2).

$$\langle \psi_m(k), \psi_n(k) \rangle = \sum_{k=-\infty}^{\infty} \psi_m(k) \psi_n^*(k), \quad (2)$$

in which $\varphi_n(k)^*$ represents the complex conjugate of $\varphi_n(k)$. To be classified as orthonormal, a function must satisfy the following requirements:

$$\begin{aligned} - \langle \psi_m, \psi_n \rangle &= 0, & \text{for } m \neq n; \\ - |\psi_n| &= 1, & \forall n; \end{aligned}$$

in which $|\varphi_n(k)| = \sqrt{\langle \varphi_n(k), \varphi_n(k) \rangle}$.

As examples of orthonormal functions, one might mention Hermite, Jacobi, Laguerre, Legendre, Kautz and the Generalized Orthonormal Basis Functions, GOBF. In this work, Kautz functions are employed.

2.2 Kautz Functions

Kautz functions are orthonormal functions parametrized by complex poles [8,9]. Equations (3) and (4) present the general form of Kautz functions:

$$K_{2m}(z) = \frac{\sqrt{(1-\tau^2)(1-\sigma^2)}}{z^2 + \sigma(\tau-1)z - \tau} \left[\frac{-\tau z^2 + \sigma(\tau-1)z + 1}{z^2 + \sigma(\tau-1)z - \tau} \right]^{m-1}, \quad (3)$$

$$K_{2m-1}(z) = \frac{z(z-\sigma)\sqrt{1-\tau^2}}{z^2 + \sigma(\tau-1)z - \tau} \left[\frac{-\tau z^2 + \sigma(\tau-1)z + 1}{z^2 + \sigma(\tau-1)z - \tau} \right]^{m-1}, \quad (4)$$

in which $m \in N$, stands for the complex variable associated with the Z transform, $K_{2m}(z)$ and $K_{2m+1}(z)$ are the even and odd Kautz functions, respectively. Considering that β and $\bar{\beta}$ are the complex conjugate poles that parametrize these functions, and can be expressed by:

$$\sigma = (\beta + \bar{\beta}) / (1 + \beta\bar{\beta}), \quad (5)$$

$$\tau = -\beta\bar{\beta}. \quad (6)$$

The use of orthonormal functions in nonlinear models aims to reduce the number of terms required by the Volterra or NARX models [8,10]. In this context, FIR models described by Kautz basis (as Volterra-OBF models) can be implemented by concatenated filters, as depicted in Figure 1.

The idea behind of NARX-OBF models came from the Volterra-OBF models. Thus, NARX-OBF can be seen as a feedback version of Volterra-OBF model [11]. Eq. (7) is the mathematical expression of a NARX-OBF model.

$$\hat{y}(k) = M_u(k) + M_y(k) + M_{uy}(k), \quad (7)$$

in which $M_u(k)$, $M_y(k)$ and $M_{uy}(k)$ stands for the input, output and hybrid model components, respectively. These components are expressed by equations (8), (9) and (10).

$$M_u = \sum_{m=1}^n c_m^u w_m^u + \sum_{p=1}^n \sum_{q=p}^n c_{p,q}^u w_p^u w_q^u, \quad (8)$$

$$M_y = \sum_{m=1}^m c_m^y w_m^y + \sum_{p=1}^m \sum_{q=p}^m c_{p,q}^y w_p^y w_q^y, \quad (9)$$

$$M_{uy} = \sum_{p=1}^m \sum_{q=1}^n c_{p,q}^{uy} w_p^u w_q^y, \quad (10)$$

in which the terms w_i^u are versions of the input, $u(k)$, filtered by a i -th order Kautz function, being $i=1, 2, \dots, n$. The terms w_j^y are versions of the output, $y(k)$ filtered by a j -th order Kautz function, being $j=1, 2, \dots, m$. Further, c_i^u (for $i=1, 2, \dots, n$), c_j^y (for $j=1, 2, \dots, m$) and c_{jl}^{uy} (for $k=1, 2, \dots, m$, and $l=1, 2, \dots, n$), are the coefficients of input, output and hybrid terms (nonlinear combination between the filtered input and output signals), respectively.

NARX-OBF models can also be expressed in the concatenated filters form, Figure 2 shows the idea. It is worth mentioning that, in this work, the NARX-OBF model is truncated in the 2nd order kernel. Once the model is defined, it is necessary to determine its parameters, in order to capture the system's dynamic whose one desire to model. In this scenario, next section is dedicated to detailing the parameter selection in NARX-OBF models.

3. Parameter Selection in NARX-OBF Models

As stated in section 1, the goal of this work is to determine the structure and parameters of an ARX-OBF model for a nonlinear system. To that end, a methodology which combines a Genetic Algorithm, GA, and the Levenberg-Marquardt, LM, method is proposed. The GA is used to find the model structure. It is worth pointing out that the GA searches for the terms that best represent the system dynamic. Thus, the algorithm finds a simplified structure for the model, aiming to reduce the computational effort.

The GA is also used in the search for the pole that parametrizes the orthonormal functions. Further, LM is the method used to find the coefficients of the model.

An appealing advantage of heuristic methods concerns stability. NARX-OBF models are feedback models and, therefore, might be unstable. Instability is a problem for conventional parameter determination methods, which may not be able to solve the estimation problem. Heuristic methods, however, are based on a population of solutions. These solutions are categorized regarding the fitness function. A solution resulting in an unstable model is poorly evaluated. Hence, the natural dynamic of the GA is able to neglect unstable models.

Next sections are dedicated to present the main ideas concerning GAs and LM method.

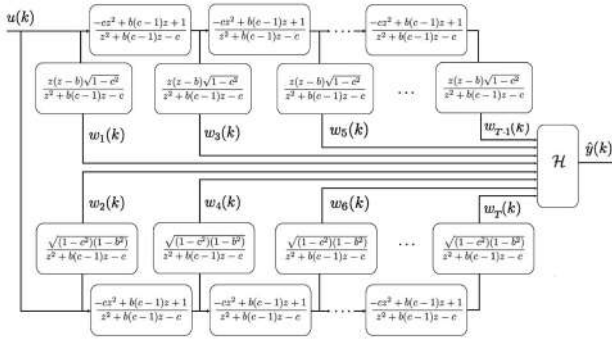


Figure 1. OBF model with Kautz dynamics^[8]. $u(k)$ is the input and $\hat{y}(k)$ is the estimated output of the system to be identified^[11]

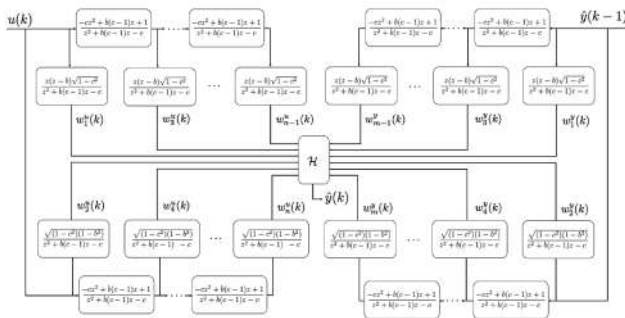


Figure 2. NARX-OBF model in the form of concatenated filters. Operations expressed by equations (8), (9) and (10) are represented by the H operator^[11]

3.1 Genetic Algorithms

In the last decades, optimization problems have motivated great improvements in mathematics and engineering. Methods like Newton, steepest descent and Levenberg-Marquardt have made possible the solution of a series of design optimization problems^[14]. However, these methods require strong conditions to have their convergence proved, such as availability of gradients and convexity^[15]. In several industrial applications the designer has to deal with some peculiarities such as nonlinearity, non-convexity, existence of several local minima, and presence of discrete and continuous design variables, among others^[16].

Optimization methods that can potentially circumvent the problems mentioned above are the heuristic algorithms. Some advantages of these algorithms include: (i) these methods do not require gradient information and can be applied to problems in which the gradient is not defined; (ii) these algorithms are not “trapped” in local minima, if correctly tuned; (iii) these methods can be applied to discontinuous functions; (iv) these algorithms provide a

set of sub-optimal solutions instead of a single solution.

Among the most popular heuristic algorithms are the Genetic Algorithms, GA^[17], the Ant Colony Optimization, ACO^[18], and the Particle Swarm Optimization, PSO^[19], all inspired by biological principles.

Genetic Algorithm, GA, was developed by John Holland in the 1960s. Inspired by Darwin’s evolutionary ideas, Holland has created a method in order to solve optimization problems that dispenses Jacobian or Hessian matrices of the problem^[17]. The GA extracts an emergent behavior of convergence. Emerging behaviors involve the application of simple rules, over and over again, which generates complex behaviors^[17].

In a GA, each individual is modeled as a set of constants $[C_1, C_2, C_3, \dots, C_n]$, called genes. These constants form a vector, C , known as a chromosome.

$$C : \begin{bmatrix} c_1 & c_2 & c_3 & \dots & c_n \end{bmatrix}$$

such constants are real for the treated problem. GA have a whole population, P , of chromosomes:

$$P = \{C_1, C_2, C_3, \dots, C_m\}, \quad (11)$$

in which each chromosome is analyzed by an evaluation function, known as a fitness function, $Fit(C): R^n \rightarrow R$, and the chromosomes with the best fitness will have greater reproducing likelihood in the next GA generations.

Genetic Algorithm is a highly parallel mathematical algorithm which transforms a population of mathematical objects, with well-defined evaluation function, $Fit(C)$, in a new population of mathematical objects, following the Darwinian principles of reproduction of the most adapted.

According to Algorithm 1, a classical GA creates a random population of solutions, expressed by P . This population consists of possible solutions, C , to the problem addressed.

Algorithm 1: Genetic Algorithm

Initializes the population of solutions, P ;
 Simulates the model generated by each chromosome;
 Fitness is calculated for each chromosome, $F_{it}(C)$;
 while $F_{it} > Stop_{criterion}$ do
 The best individual is saved;
 Selecting parents;
 Apply Crossover Operator;
 Apply Mutation Operator;
 Simulate the model corresponding to each chromosome;
 Evaluate Fitness function for each chromosome;
 end while

The solutions are, then, evaluated by the function $Fit(C)$. After these steps, the best individuals (chromosomes with the lowest image in the evaluation function, Fit) are (more likely) selected to be progenitors of the next generation.

The Crossover and Mutation operators are applied to the population and the generated individuals are evaluated. This cycle is repeated until the stop criterion is reached, i.e., the fitness value of the best chromosome is smaller than a certain threshold: $Fit(C) < Stop_{criterion}$.

It should be emphasized that GAs do not guarantee convergence to the optimum of the problem, and may end up confined to a local region^[20]. A point x_l is a local minimum if there is a neighborhood v (of x_l), such that $f(x_l) \leq f(x)$ for $x \in v$ ^[14].

3.1.1 Crossover

The Crossover operator was inspired by the biochemical process of Crossing-Over, in which parts of two chromosomes are exchanged in the process of sexual reproduction^[20]. Figure 3 shows the Crossover operator action under chromosomes $f=[f_1, f_2, f_3, \dots, f_n]$, and $g=[g_1, g_2, g_3, \dots, g_n]$. This operator performs the local search in the search space^[17].

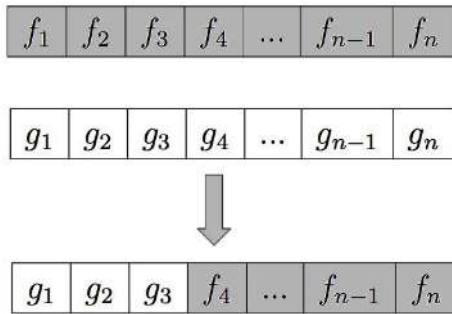


Figure 3. Example of Crossover operator

3.1.2 Mutation

The Mutation operator can perform an exploration (in the searching space) by inserting a random constant into a random gene position, as expressed in Figure 4.

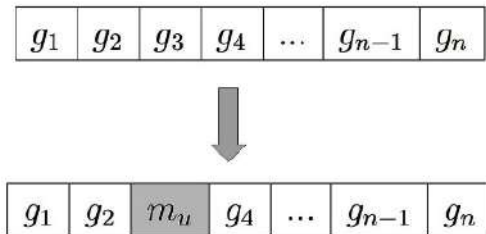


Figure 4. Example of Mutation operator

This operator has essentially the global search function, being complemented by the crossover operator, that performs local searches, composing the mechanism of a classical Genetic Algorithm.

3.2 Levenberg-Marquardt

The Levenberg-Marquardt method can be derived by substituting the exact line search strategy (see^[15]), for the Confidence Regression strategy (see^[14]). The use of the Confidence Regression strategy avoids the main problem of the Gauss Newton method, which occurs when the Jacobian, $J(x)$, stop being full rank, or near to it^[14]. Generally, the Hessian of a generic function, $f(x)$, can be approximated as:

$$\nabla^2 f(x) \approx J_{\xi}(x)^T J_{\xi}(x), \quad (12)$$

In order to simplify (12), $\nabla^2 f(x)$ can be expressed by $B(x)$, as (13).

$$B(x) = J_{\xi}(x)^T J_{\xi}(x) \approx \nabla^2 f(x). \quad (13)$$

The idea of the LM method is to disturb the matrix $B(x)$, considering $B(x) + \rho I$, for $\rho > 0$. This method can be understood as the Gauss-Newton method with the following modification:

$$\begin{cases} x_{k+1} = x_k + \Delta x_k \\ \Delta x_k = -[J_{\xi}(x)^T J_{\xi}(x) + \lambda I]^{-1} [J_{\xi}(x)^T \xi(x)], \end{cases} \quad (14)$$

in which $\lambda \in \mathbb{R}$ and I is the identity matrix.

It is reasonable to consider the use of hybrid algorithms. Such algorithms behave as LM, for small residues, and apply the Newton method, for larger residues^[14]. As the Gauss-Newton methods, LM is based on an expansion into Taylor's series^[21]. The search mechanism used by the Levenberg-Marquardt method can be observed in the algorithm 2.

Algorithm 2: Levenberg-Marquardt Algorithm.

```

Let be  $x_0 \in \mathbb{R}^n$  ;
Calculate  $d_0$ , solution of:
 $[B(x_0) + \lambda I] \Delta x_0 = -\nabla f(x_0)$ ;
in which:  $B(x) = J_{\xi}(x)^T J_{\xi}(x)$ ;
 $x_1 = x_0 + \Delta x_0$  ;
k=1;
while  $\nabla f(x_k) \neq 0$  do
    Calculate  $\Delta x_k$ , solution of:
     $[B(x_k) + \lambda I] \Delta x_k = -\nabla f(x_k)$ ;
    Determine  $x_{k+1}$ ;
     $x_{k+1} = x_k + \Delta x_k$  ;
    k=k+1

```

3.3 Proposed Methodology

NARX-OBF models, in their complete form, have a high number of terms^[8,9], therefore, reducing the order of the model is interesting from a computational perspective.

In order to reduce the model order, the GA fitness function take two aspects under consideration: (i) the number of terms, N_C ; and (ii) the minimization of the MSE . Eq. (15) expresses the fitness function used in this work, which was inspired by the Akaike criterion^[12].

$$f_{it}(MSE, N_C) = N \times MSE + 0.1 N_C \times \log(N), \quad (15)$$

in which N is the number of samples in the input and output signals of the system, MSE is the mean square error, N_C stands for the number of coefficients involved in the model and the multiplier 0.1 is an empirical coefficient, used to balance the weight of terms.

The genes used in the GA are depicted in Figure 5. The real and imaginary parts of the Kautz function pole and the presence of terms coefficients in the NARX-OBF model are genes in the GA chromosome. Thus, the evolutionary dynamics of the GA is responsible for selecting the terms of the model which are representative for the system to be identified, performing an order reduction of the NARX-OBF model.

$Real(\beta)$		$Imag(\beta)$	
p_1	p_2	$\dots$	p_n

Figure 5. Structure of genes that compose a chromosome in the proposed GA. is the pole of the Kautz functions and the genes of the vector represent the presence or absence of each term of the NARX-OBF model, in its simplified version

Note: If, e.g., p_1 is 0 the first term of the NARX-OBF model is disregarded. If p_2 is 1, the second term of the series is maintained.

In the proposed methodology the idea is interleaving a heuristic algorithm, GA, and a deterministic one, LM algorithm. Mixing these two algorithms, one can achieve the advantages of heuristic algorithms (do not get stuck in local minima) and the advantages of deterministic algorithms (the guaranty of finding the global minimum in a concave region of the search space). In this context, GA is responsible for finding: (i) the orthonormal functions pole; and (ii) the NARX-OBF model structure, whereas the LM is responsible for finding the model coefficients. The loop interaction between the two algorithms is illustrated in Figure 6.

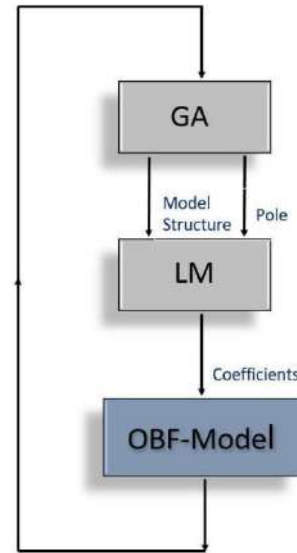


Figure 6. Search mechanism of OBF-model parameters

In order to testing this methodology, in the next section, it will be presented the identification process of a non-linear system.

4. Identification of a Magnetic Levitator

In order to validate the proposed method for reducing the order of a NARX-OBF model, a magnetic levitation system was chosen and identified. The system consists of 2 permanent magnets and a mobile magnetic disk. Four coils, operated two at a time, are able to control the disk movement. In this paper, the model is obtained concerning the x-axis. That is, the movements in the axes y and z are neglected^[22]. A schematic of the system is depicted in Figure 7.

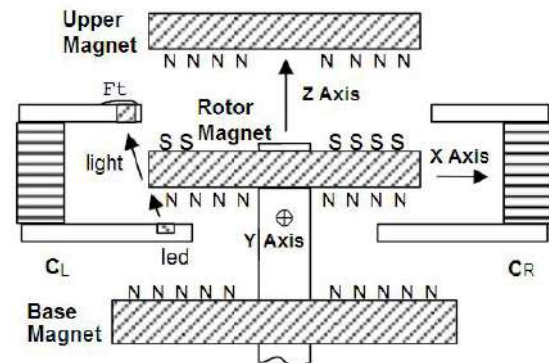


Figure 7. Magnetic levitator schematic, adapted from^[22]

The identification of a nonlinear system, such as the one in Figure 7, starts by the application of a Persistently Exciting, PE, signal to its input. A signal is said to be per-

sistently exciting if, considering the need to estimate N_p parameters, it has spectral power in N_p bands of frequency^[4].

A widely used kind of PE signal is the Pseudo-Random Multi Level Signal, PRMLS. The variable range of the signal amplitude is desirable in the identification of nonlinear systems, since it provide the excitation of the system several dynamics^[3].

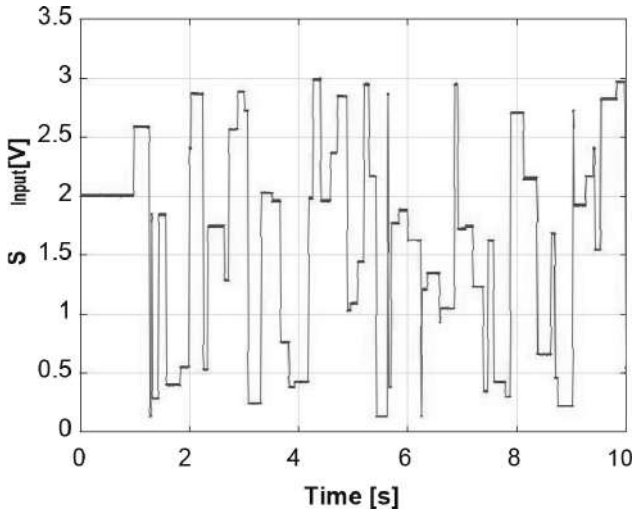


Figure 8. Input data sampled in the magnetic levitator

According to Earnshaw's theorem^[23], the system in Figure 7 is unstable. Thus, in order to circumvent the instability problem, the system operates in closed loop under the action of a Proportional Integral, PI, controller.

Figure 9 depicts the system output response to the input signal (presented in Figure 8). Both signals, input and output, are composed by 100,000 samples. The sampling period is of 1 ms. Moreover, in the validation of the model, 20,000 samples were used.

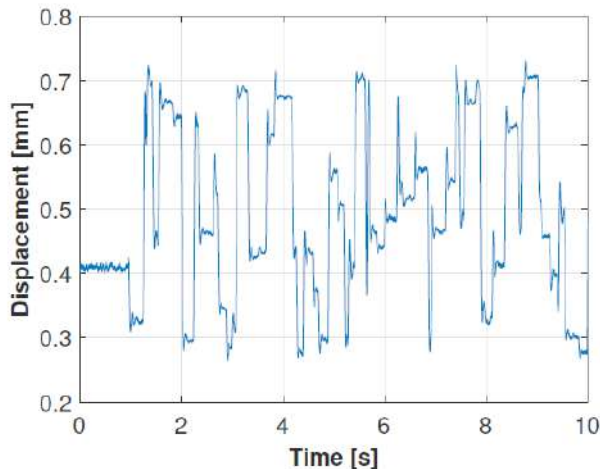


Figure 9. Output data sampled in the magnetic levitator

Table 1. Genetic Algorithm Parameters

Parameter	Value
Population	200 Chromosomes
Selection Method	Tournament
Mutation Rate	varies linearly (5 to 20 %)
Crossover Rate	varies linearly (80 to 40 %)

Table 1 shows the specifications of the GA, which performed the parameter search for the NARX-OBF model. It is worth to emphasize that the metric applied in the identification process aims the minimization of the *MSE* as well as the reduction of the model complexity, as expressed in Eq. (15).

Genetic algorithms have a stochastic component^[17]. Thus, there is no guarantee that the *MSE* has reached its global minimum. However, if the GA is well tuned, it is possible to find reasonable parameters. Further, in this work, the algorithm applies the GA to search the model structure and the OBF pole, and applies Levenberg-Marquardt method to find the model coefficients, interweaving the methods. It is important to mention that the GA loop keeps going on until the stop criterion is reached, i.e., the *MSE* of the best solution reach a value smaller than ϵ . The complete structure of this hybrid GA can be seen in Figure 10.

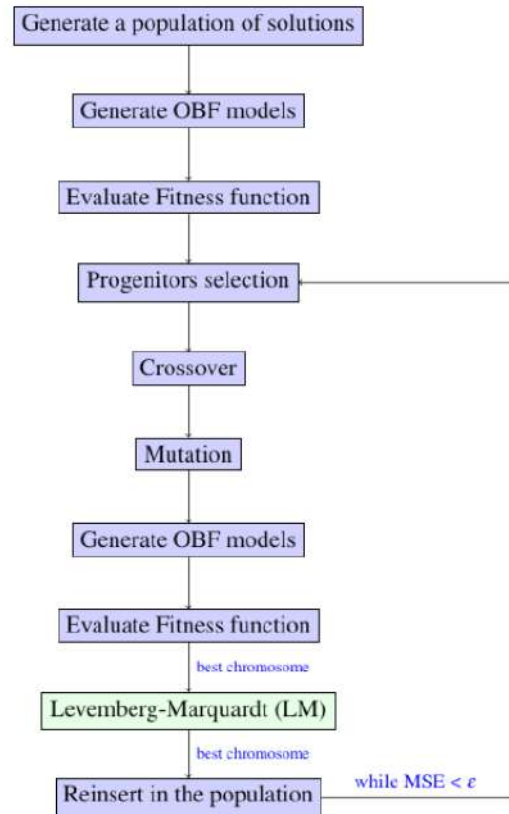


Figure 10. The proposed Genetic Algorithm structure

expressed as a flowchart, ϵ is a higher limit for the MSE

Next section is dedicated to present the main results obtained in the identification of the magnetic levitator.

5. Results

After the closed-loop identification of the magnetic levitator, the *MSE* obtained in each complete NARX-OFB model, with different numbers of orthonormal functions, is shown in Table 2. In this table, N_F stands for the number of orthonormal functions used, and N_C corresponds to the number of coefficients for each NARX-OFB model. The larger N_C , the greater is the number of terms in the model. Therefore, models with larger N_C are more time consuming, computationally speaking.

Table 2. MSE for Complete NARX-OFB Models

N_F	N_C	Pole	MSE
2	14	$0.6479 \pm 0.3812i$	1.9416×10^{-3}
4	44	$0.5521 \pm 0.4012i$	9.7212×10^{-4}
6	90	$0.6017 \pm 0.0686i$	5.4947×10^{-6}

The *MSE* for the NARX-OFB models with order reduction, and their fitness values, can be seen in Table 3.

Table 3. MSE for simplified NARX-OFB models

N_F	N_C	Pole	MSE	Fitness
2	8	$0.3833 \pm 0.7870i$	1.8370×10^{-3}	40.1808
4	11	$0.4770 \pm 0.5850i$	3.5540×10^{-3}	11.8331
6	54	$0.5487 \pm 0.0289i$	3.5256×10^{-4}	30.2768

Figures 11, 12 and 13 illustrate the time responses of the reduced order NARX-OFB models cited in Table 3.

In order to make a visual comparison, Figure 14 depicts the performance of the complete NARX-OFB model, mentioned in Table 2.

Comparing tables 2 and 3 one can see that removing some terms of the complete NARX-OFB model does not represent a significant *MSE* increasing. Furthermore, analyzing the first row of tables 2 and 3, it is possible to see that removing 6 terms of the complete model leads to a small reduction in the *MSE*. This result shows that not all terms of the complete model are in accordance with the system dynamic. Therefore, removing these terms has small impact in the *MSE*. Thus, some results obtained by order reduction of NARX-OFB models can approximate complete NARX-OFB models without loss of generality.

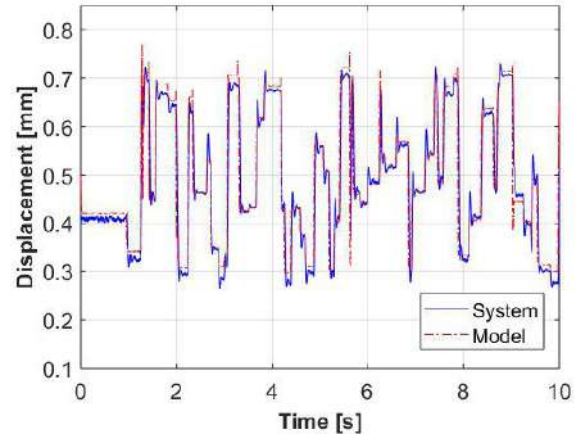


Figure 11. Identification results for the magnetic levitator, with reduced order NARX-OFB, using 2 Kautz functions and 8 coefficients (terms)

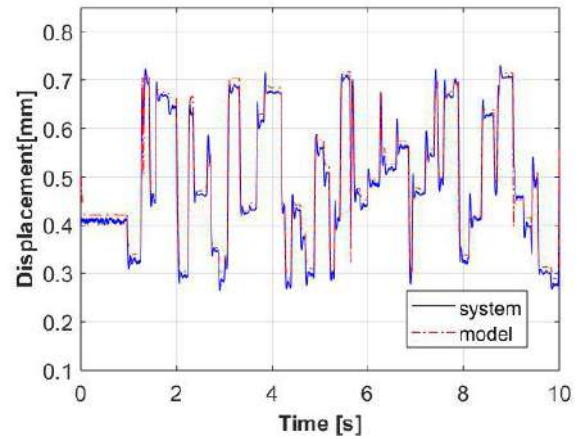


Figure 12. Identification results for the magnetic levitator, with reduced order NARX-OFB, using 4 Kautz functions and 11 coefficients (terms)

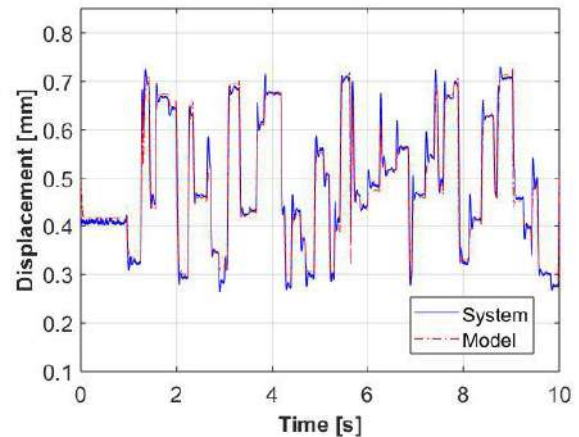


Figure 13. Identification results for the magnetic levitator, with reduced order NARX-OFB, using 6 Kautz functions and 54 coefficients (terms)

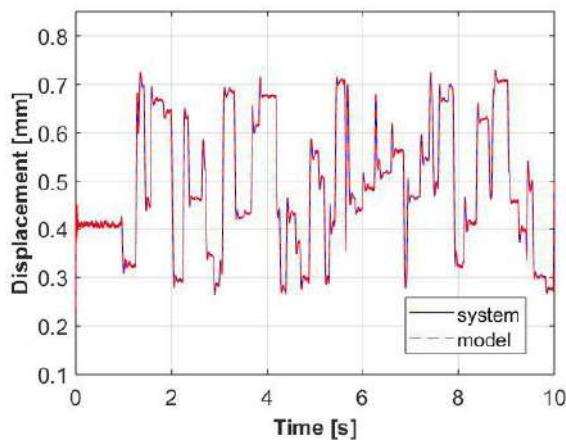


Figure 14. Identification results for the magnetic levitator, with complete NARX-OBF, using 6 Kautz functions and 90 coefficients (terms)

The time that each simplified model, expressed in Table 3, took to be simulated is shown in Table 4. The simulation time was computed for 10 simulations of each model, the average of the simulation time is expressed in the final row of Table 4. In this table, K indicates the number of Kautz functions and C, the number of coefficients. Thus, the model 2K 8C stands for the model with 2 Kautz functions and 8 coefficients, which is in the first row of Table 3.

Next section presents the main conclusions of this work.

Table 4. Simulation time for simplified NARX-OBF models

Time	2K 8C [s]	4K 11C [s]	6K 54C [s]
1	0.1124	0.2796	0.3806
2	0.1039	0.2535	0.3599
3	0.1057	0.2300	0.3521
4	0.1284	0.3708	0.4583
5	0.0905	0.2021	0.3107
6	0.0885	0.1995	0.3089
7	0.8889	0.2032	0.3075
8	0.0903	0.2008	0.3040
9	0.0892	0.1949	0.3074
10	0.0888	0.2027	0.3059
Average Time	0.0906	0.2032	0.3107

6. Conclusion

This paper proposes a method to obtain the order reduction of a NARX-OBF model. A Genetic Algorithm combined with the Levenberg-Marquardt method is used to find the model structure and parameters. The validation of

the proposed methodology was based on the closed-loop identification of a magnetic levitator.

The results shown in tables 2 and 3 allow the conclusion that the order reduction is not only possible, but has little impact in the *MSE* value. Thus, one might say that some of the terms which compose the complete NARX-OBF model can be suppressed without lost the capacity of representing the system behavior. Moreover, in Table 3 it is possible to observe that the best fitness value is found in the intermediate situation, between the minimum *MSE* and the minimum number of terms, N_c . This case portrays the optimization of both criteria at the same time.

It is important to mention that GA is a probabilistic method and there is no guarantee in achieving the best *MSE* in the identification process. However, in average, it is possible to find reasonable parameters, as shown in tables 2 and 3.

The next steps of this work include the use of Genetic Programming to select the candidates to compose the simplified NARX-OBF model and the use of GOBFs (Generalized Orthonormal Basic Functions) instead of only Kautz functions.

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ARTICLE

A Novel Process for SiGe Core-Shell JAM Transistors Fabrication and Thermal Annealing Effect on Its Electrical Performance

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ABSTRACT

In this study, we fabricate Si/SiGe core-shell Junctionless accumulation mode (JAM) FinFET devices through a rapid and novel process with four main steps, i.e. e-beam lithography definition, sputter deposition, alloy combination annealing, and chemical solution etching. The height of Si core is 30 nm and the thickness of Si/SiGe core-shell is about 2 nm. After finishing the fabrication of devices, we widely studied the electrical characteristics of poly Si/SiGe core-shell JAM FinFET transistors from a view of different L_g and W_{ch} . A poly-Si/SiGe core-shell JAMFETs was successfully demonstrated and it also exhibits a superior subthreshold swing of 81mV/dec and high on/off ratio $> 10^5$ when annealing for 1hr at 600°C. The thermal diffusion process condition for this study are 1hr at 600°C and 6hr at 700°C for comparison. The annealing condition at 700°C for 6 hours shows undesired electrical characteristics against the other. Results suggests that from over thermal budget causes a plenty of Ge to precipitate against to form SiGe thin film. Annealing JAMFETs at low temperature shows outstanding Subthreshold swing and better swing condition when compared to its counterpart i.e. at higher temperature. This new process can still fabricate a comparable performance to classical planar FinFET in driving current.

1. Introduction

Complementary metal-oxide-semiconductor (CMOS) device have been the dominant device for ultra large -scale integration (ULSI) in semiconductor industry for decades due to its high speed and low power consumption. In recent years, owing to the limitation in miniaturization of VLSIs, we are looking for a novel material introduction to replace the planar Si channel devices in order to achieve high performance. Alternate channel materials such as Ge and SiGe are in great interest due to their higher mobility than Si. Moreover,

stereoscopic channel structure are the primary methods to advance performance.

Si/SiGe core-shell hetero-structures are recognized as the most promising solutions to further continue the Moore's law beyond conventional planar bulk technologies are in great interest for p-type high-performance MOSFET. Core/shell structures with either Si or Ge as the core or shell have been researched such as Ge/Si^[1], SiGe/Si^[2], or Si/Ge^[3] core/shell NW hetero-structures have been proposed to improve the hole transport in p-type FETs. Compared to traditional 2D Si planar devices, core-shell hetero-structure devices offer higher drive current

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and strong gate controllability due to its geometrical advantage. A core-shell MOSFET fabrication approach passes through two-step process. First, grow core structure through bottom-up^[2-4] or top-down^[1,5] methods. Second, the shell structure around the core is grown by chemical vapor deposition (CVD). However, there exist a challenge in this method that hetero-epitaxy is very difficult in the case of horizontal layers^[6].

In this study, we fabricate core-shell devices through a rapid and novel process with four steps, i.e. e-beam lithography definition, sputter deposition, alloy combination annealing, and chemical solution etching. Different from above methods, we introduce a whole low cost and low thermal budget methods for core-shell structure fabrication a new choice. With the miniaturization of the channel length, the diffusion problem of short-channel junction doping occurs and it is difficult to accurately control the abrupt junctions^[7]. Thus, the junctionless (JL) FETs have been proposed to solve the problem in the fabrication of ultra-shallow and abrupt junctions profile^[8-10]. Different from inversion-mode (IM) FETs, because channel and S/D of junctionless (JL) FETs have the same heavily doping concentration and doping types, there is no PN junctions exists between channel and S/D. Furthermore, (JL) FETs have different conduction mechanism compared to the inversion-mode (IM) FETs.

Figure 1 shows the cross-sectional structures of p-type FETs along the S/D direction showing the doping profile, including (a) inversion mode (IM) FETs, (b) junctionless (JL) FETs^[9,10]. Owing to thermal diffusion of S/D dopants into the channel, (IM) FETs has a shorter effective channel length than the physical channel length. For the (JL) FETs, because the work function difference between the gate electrode and the channel pushes away the depletion region from gate edge to the S/D, it has a longer effective channel length than the physical channel length^[11]. Hence, (JL) FETs are regarded as having stronger immunity against short-channel effects than (IM) FETs^[12-14]. Because of the large number of carriers, there is a big challenge in the (JL) FETs process that carriers in the channel are difficultly fully depleted. For this reason, the channel dimension of (JL) FETs must be a small cross-sectional area. However, (JL) FETs have a critical issue which is high S/D parasitic resistance due to the small channel dimension and random dopant fluctuation (RDF)^[15]. To overcome these problems, junctionless accumulation mode (JAM) FETs have been proposed. Junctionless accumulation mode (JAM) FET with additional S/D implantation have been implemented for lowering the S/D parasitic resistance^[9,16,17], as shown in Figure 2. Different from conduction mechanism of (JL) FETs, when devices turn on,

(JAM) FETs have an accumulation layer at the channels surface^[9].

In this study, we proposed a (JAM) FETs but without channel doping, the SiGe channel is intrinsic. And we depend S/D implantation with B¹¹⁺ to demonstrate a p-type junctionless accumulation mode FETs. After introducing several concepts above, we can briefly clarify the target to effectively achieve novel way of process for fabricating SiGe Core-Shell transistors.

2. Experimental Procedure

In this study, we demonstrate a Si core clad with a SiGe shell JAMFET, there are several methods to fabricate Si core / SiGe shell structure, such as SiGe condensation^[6,18] or directly epitaxy SiGe layer on the Si fins^[19]. Here, we choose DC sputter to deposit Ge on the Si fins. The reason why we choose sputter deposition to fabricate core-shell is that sputtering has advantages such as high purity and vacuum, furthermore, it can form a thin film in low temperature.

Main process flow is shown in Figure 3. Device fabrication begins on a 6-inch Si wafer grown with 200 nm wet oxide, after that, a 50-nm-thick Si₃N₄ layer is deposited on it. Then the 40-nm-thick undoped amorphous Si (α -Si) layer is deposited via low-pressure chemical vapor deposition (LPCVD), then α -Si is transformed to poly-Si through solid-phase crystallization (SPC) for 24 hrs at 600°C in N₂ ambient.

The active region is defined by E-beam stepper and reactive-ion etching (RIE) to form a poly-Si fin-channel. Then, 12-nm-thick amorphous Ge (α -Ge) layer is deposited by sputter and 20-nm-thick PESiO₂ is deposited on amorphous Ge layer to prevent Ge from oxidation during the thermal process. The combination of amorphous Ge between poly-Si is via the horizontal furnace tube in different annealing temperature and time i.e. 1 hr at 600°C and 6 hr at 700°C to form the different Ge content in SiGe channel. However, the Ge completely covers whole wafer during sputtering. Because the etching selective ratio between SiGe and Ge is different, we depend on wet etching method to remove the residual Ge on the Si₃N₄ layer. We choose the etching solution from a few papers^[20,21] and the suitable choice is (HCl+H₂O₂) : H₂O 0.5% at the room temperature. As mentioned in process flow, the poly-Si core/poly-SiGe shell structure channel is fabricated. Next, the gate oxide of 7-nm-thick Al₂O₃ is deposited by ALD and the TiN is deposited immediately as gate metal. After the gate patterning, B¹¹⁺ implantation (1×10^{15} cm⁻² at 10 keV) was carried out to form p-type S/D and the dopant activation is done through microwave annealing and the schematic configurations of key process steps for the po-

ly-Si / SiGe core-shell JAMFETs are shown in Figure 4. Finally, material and electrical performance of poly-Si/SiGe JAMFET were analyzed and discussed for two thermal annealing condition i.e. 1 hr at 600°C and 6 hr at 700°C.

3. Results and discussion

Figure 5 shows the depth profile of active region after the SiGe combination annealing by the X-ray photoelectron spectroscopy. As shown in Figure 5, the SiGe shell composition can be observed, the content of Ge at the channel surface are 20.8%, 36.4% and 32.9% for different annealing condition (1hr at 600°C, 1hr at 700°C and 6hrs at 700°C), respectively. In addition, no matter which annealing condition is, the profile of Ge has higher content at the surface forming a SiGe alloy. When etching depth reaches a deeper position, the Ge content decrease obviously about below 5%. In this paper, we will mainly perform observe electrical performance at annealing condition for 1hr at 600°C and 6hrs at 700°C. It is noteworthy that when the annealing temperature is 700°C, more Ge penetrating into the Si layer than annealing temperature is 600°C. Hence, the Ge penetration is severe for higher annealing temperature. Finally, SiGe is detected on the shell of this structure through energy dispersive spectrometer (EDS). From Figure 6 we can demonstrate that the element content on the shell of silicon and germanium is 85.35% and 14.65%.

In this part, we investigate the material analysis results and electrical parameters of poly-Si/SiGe core-shell JAMFETs with different SiGe combination annealing conditions. The measured dimensions of channel width (W_{ch}) are 40 nm, 60 nm, 80 nm and 100 nm and gate length (L_g) are 60 nm, 80 nm, 100 nm, 120 nm, 150 nm, 200 nm and 400 nm. Figure 7 shows the cross-sectional transmission electron microscope (TEM) image of poly-Si/SiGe core-shell JAMFET. In Figure 7, a rectangular shape core-shell structure is successfully fabricated in which channel width (W_{ch}) $\times$ channel height (H_{ch}) is (68 $\times$ 30) nm and effective width (W_{eff}) is 128 nm. We observe that the white region is a 2.17 nm-thick thin SiGe layer cladding on a 30 nm-thick poly Si layer after chemical solution etching and a 5.16 nm-thick Al_2O_3 layer and a 63.25 nm-thick TiN surround the core-shell channel.

We expect that the specific condition of annealing can combine Ge and Si at the channel surface but rarely penetrates Ge into Si. There are mainly two SiGe combination annealing condition i.e. 1hr at 600° and 6hrs at 700°C in N_2 ambient. The sample structure is poly $Si_{1-x}Ge_x$ ($x = 20\% \sim 37\%$) / poly Si / wet SiO_2 / substrate. Furthermore, we investigate the electrical characteristics

such as I_D versus V_G , threshold voltage, subthreshold swing, DIBL and on current of the condition of Poly-Si/SiGe core-shell JAMFETs when SiGe combination annealing condition is 1hr at 600°C and 6hrs at 700°C with different dimension. The electrical performance of poly-Si/SiGe core-shell JAMFETs were measured at room temperature by semiconductor device analyzer (KEITHLEY, version V9.1 SP3). Figure 8 shows the outstanding transfer characteristics of poly-Si/SiGe core-shell JAMFET with channel width = 40nm and gate length of 100nm for different annealing activation time. i.e. for 1hr at 600°C and 6hr at 700°C, respectively. The p-type Poly-Si / SiGe core-shell devices for 1hr annealing at 600°C perform a superior S.S. in comparison to the annealing for longer duration of annealing at 700°C and the resulting value is 81 mV/dec and 101 mV/dec, respectively and I_{ON}/I_{OFF} ratio is $> 10^5$. It indicates that there is improvement in sub-threshold swing when annealing for less annealing duration and temperature. Subthreshold swing was relatively higher in case of high annealing condition of Si/SiGe core shell JAMFET device. This may be due to the relatively higher interface trap density in SiGe channel when annealing at high temperature. Figure 9 displays the on-state current for p-type Poly-Si / SiGe core-shell JAMFETs as function of different channel width and fixed gate length = 100nm, where gate voltage (V_g) was varied from 0 to -2.4V with a step of -0.6V. The resulting drain current is higher in case of annealing for 6hr at 700°C when compared to its counterpart i.e. annealing for 1hr at 600°C. In an ideal MOSFET, I_d is expected to be linearly dependent on V_{gs} , as shown by equation (1).

$$I_d = C_{ox} \mu \frac{W}{L} \left[(V_{gs} - V_t) V_{ds} - \frac{V_{ds}^2}{2} \right] \quad (1)$$

Where, I_d = Drain current, C_{ox} = Oxide capacitance.

To extract V_t , an often-used method is the transconductance method, where the improved transconductance g_m is given by:

$$g_m = \frac{W}{L} \frac{2a}{q} C_{OX}^2 (V_{gs} - V_t) V_{ds} \quad (2)$$

Where; a = common base current gain, q = magnitude of electron charge.

Figure 10 and Figure 11 shows I_D vs V_G characteristics of the poly-Si / SiGe core-shell JAMFET at various fin width and fixed gate length of 100nm for 1hr at 600°C and 6hr at 700°C. The figure shows that there is no strong dependence between on current as the channel

width varies and the gate length except for that the minimum device of $W_f = 60$ nm demonstrating lower on current. By comparing the two figures, we know that the V_t roll-off phenomenon due to short channel effect is much more severe on the device for longer duration of thermal annealing condition. Furthermore, the sub-threshold properties and on-off current ratio are also degraded when the channel width increases. Figure 12 shows the distribution of subthreshold swing (SS) as function of gate length of 100nm and various channel width for 1hr at 600°C and 6hr at 700°C, respectively. It reveals that as the channel width increases from 40nm to 100nm, the subthreshold swing also increases somewhat linearly. The minimum subthreshold swing was observed at channel width of 40nm for 1hr at 600°C and the maximum swing was observed at channel width of 100nm for 6hr at 700°C, indicating the degradation of gate controllability. Subthreshold swing remains almost constant and low in the annealing condition of 1hr at 600°C for channel width of 40nm and 60nm. In contrast, the electrical performance of neutral beam etched devices is similar within the gate length region of 150 ~ 400 nm, indicating better control of the interface properties. Figure 13 shows the dependence of threshold voltage a function of different gate length at a fixed fin width=60nm under two different annealing condition for poly-Si/SiGe core-shell JAMFETs. Figure 13(a) shows the short channel effect in terms of decreasing threshold value as it is annealed at low annealing duration for 600°C from and the highest threshold voltage is about 0.299V at $W_{ch} \times L_g$ of 60 nm x 120 nm and the lowest threshold voltage is about -1.72V at $W_{ch} \times L_g$ of 60 nm x 200 nm.

From Figure 13(b), We observe that the rough trend of threshold voltage is decrease with gate length becoming long shows the short channel effects. The highest threshold voltage is about 1.53V at $W_{ch} \times L_g$ of 60 nm x 80 nm and the lowest threshold voltage is about -2.95V at $W_{ch} \times L_g$ of 60 nm x 400 nm. It is noticeable that the increase in gate length of channel material results in decrease on V_{th} of JAMFETs. Figure 14 shows the dependency of ON current on gate length of poly-Si/SiGe core-shell JAMFET at channel width of 40nm for 1hr at 600°C and 60nm for 6hr at 700°C, respectively. With the decrease in fin width, generally a slight increase in threshold voltage and a decrease in on current can be observed. In a thinner body, there are fewer amounts of inversion charge, and this is observed as a decrease in sub-threshold current and an increase in threshold voltage. With regards to the decrease in on current, it is directly related to the decrease in channel width, as can be referred in equation 1. For higher annealing temperature and longer duration, On

current slightly decreases with increase in gate length and the similar phenomena is observed for lower annealing temperature. From Figure 15 we can recognize that Ge is precipitated due to over thermal budget treatment in 6hrs 700°C annealing process. Our poly-Si/SiGe core-shell JAMFETs have shown quite good channel width scalability at low temperature thermal annealing when compared to longer annealing criterion. We realized a Si core cladded with a SiGe shell MOSFET which is fabricated with DC sputter to deposit Ge on the Si fins and through the horizontal furnace annealing in N_2 ambient and chemical etching process to remove residual Ge. In this study, driving current of silicon germanium for these devices is boosted. Although the V_{th} displays there are still have SCE in these devices.

4. Conclusions

In this study we have demonstrated a Poly-Si /SiGe core-shell structure and investigated the electrical characteristics of Poly-Si /SiGe core-shell JAMFETs. The experimental results can be confirmed in the cross-sectional transmission electron microscope (TEM) image and energy dispersive spectrometer (EDS). A Poly-Si / SiGe core-shell gate stack is displayed in cross-sectional transmission electron microscope (TEM) image. There is a very thin SiGe layer cladding the Si fin forming a Si/SiGe core-shell structure. The results of this study provide demonstration of novel technologies as well as physical insight into their performance. Studying in the terms of the electrical performance of Ion and S.S., we can observe that SiGe combination in the condition of 1hr 600°C precisely transfer and effectively enhance the electrical characteristics. The JAMFETs with channel width of 40nm and gate length of 100nm for 1hr at 600°C demonstrates the outstanding subthreshold swing of 81mV/dec and on/off ratio $>10^5$. Compare to above results, the Ion of SiGe combination in the condition of 6hrs 700°C doesn't show the behavior which SiGe can strengthen the drive current. Also, the value of SS of SiGe combination in the condition of 6hrs 700°C is higher than previous conditions. Furthermore, we introduced a whole low cost and low thermal budget methods for core-shell structure fabrication as a new option.

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Appendixes

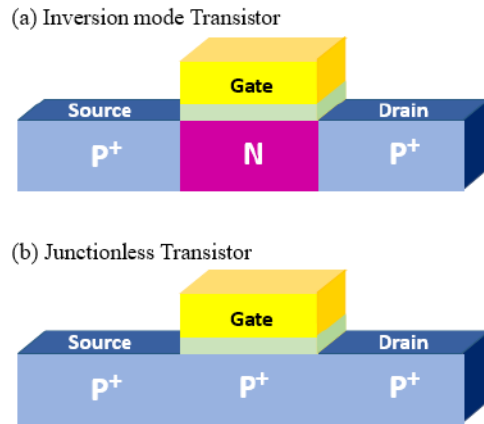


Figure 1. Cross-sectional structures of p-type FETs along the S/D direction (a) inversion mode (IM) FETs, (b) junctionless (JL) FETs

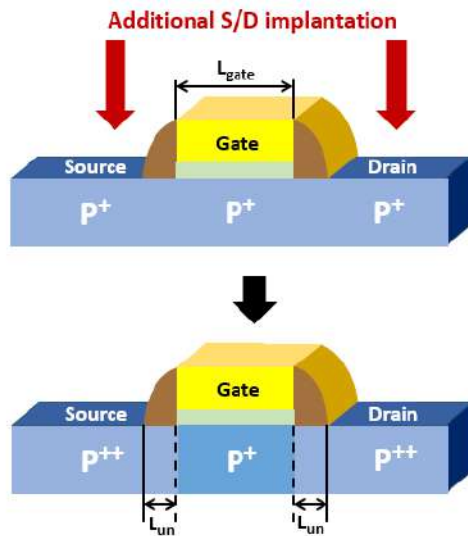


Figure 2. Junctionless (JAM) FET with additional S/D implantation

- Depositing SiO₂ 200nm on bulk Si
- Depositing Si₃N₄ 50nm on SiO₂ layer
- Depositing α -Si 40nm by LPCVD
- SPC (600°C, 24hrs)
- Active region definition
- Sputtering Ge 12nm on poly-Si
- SiGe combination
- 600°C_1H / 700°C_1H / 700°C_6H
- Wet etching Ge / Si core-SiGe shell formation
- [HCl:H₂O₂(10:1)]:H₂O 0.5%
- Al₂O₃ 7nm as gate oxide / TiN as metal gate
- Gate patterning
- Source/drain implantation
- B¹¹⁺/10keV/1E15
- RTA activation

Figure 3. Process flow of the poly-Si / SiGe core-shell JAMFETs

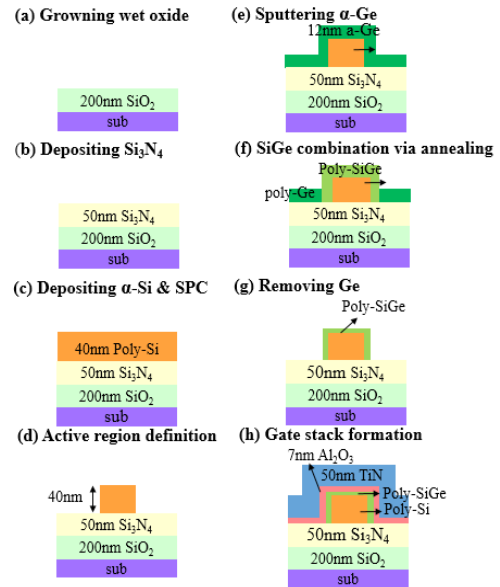


Figure 4. Schematic configurations of key process steps for the poly-Si / SiGe core-shell JAMFETs

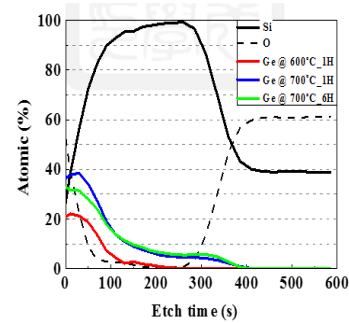


Figure 5. The depth profile of active region with three SiGe combination annealing condition, i.e. 600°C_1hr, 700°C_1hr, 700°C_6hr

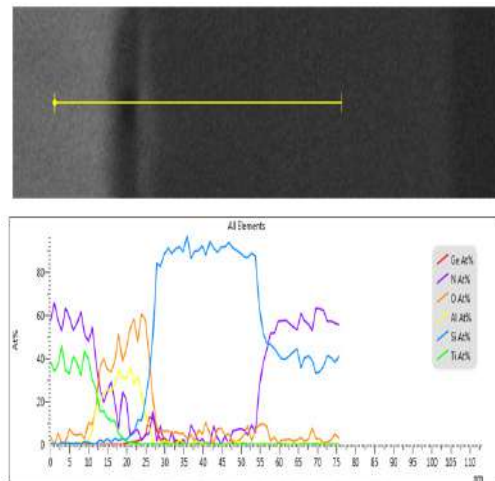


Figure 6. The element content distribution of poly-Si / SiGe core-shell JAMFETs by energy dispersive spectrometer

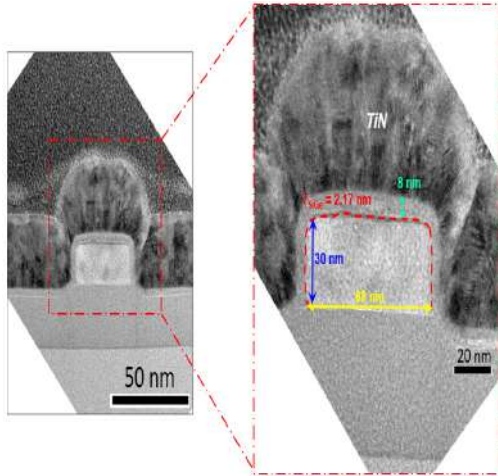


Figure 7. Cross-sectional TEM image of poly-Si / SiGe core-shell JAMFETs along the gate (b) The enlarged TEM image with $H_{ch} = 30$ nm, $W_{ch} = 68$ nm

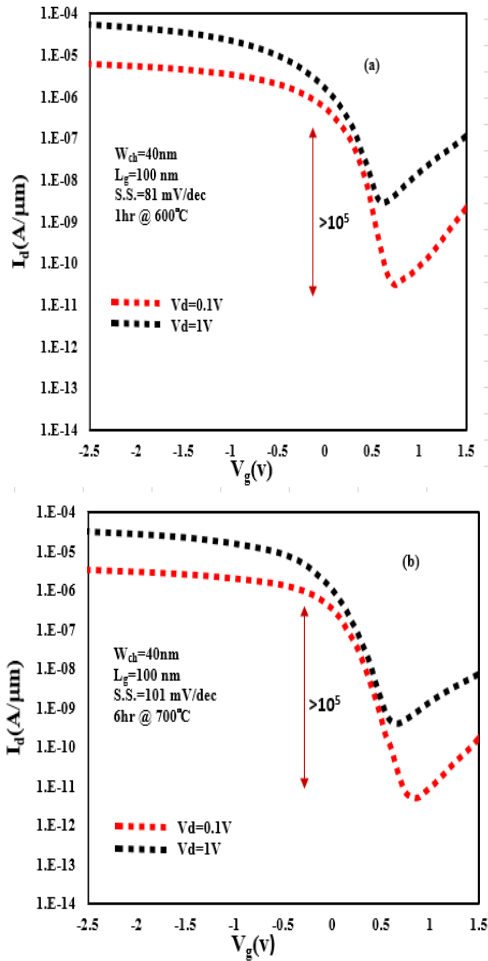


Figure 8. The outstanding transfer characteristics of the poly-Si / SiGe core-shell JAMFET at (a) W_{ch} of 40 nm and L_g of 100 nm for 1 hr at 600°C (b) W_{ch} of 40 nm and L_g of 100 nm for 6 hr at 700°C, respectively

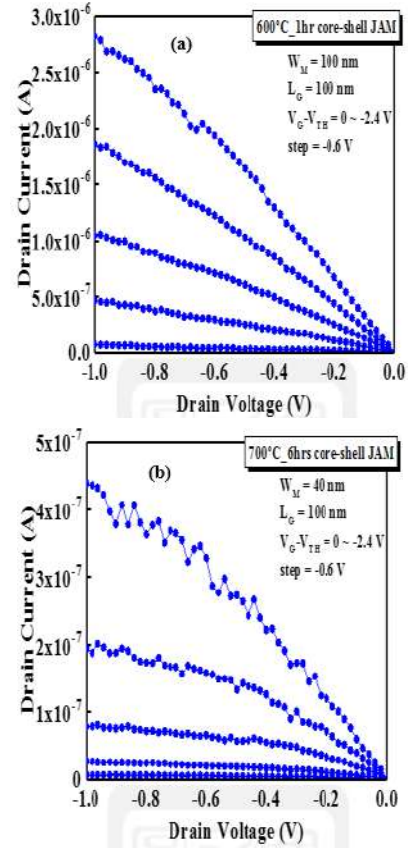


Figure 9. The outstanding I_D - V_D of the poly-Si / SiGe core-shell JAMFET at (a) W_{ch} of 100 nm and L_g of 100 nm for 1 hr at 600°C (b) W_{ch} of 40 nm and L_g of 100 nm for 6 hr at 700°C, respectively

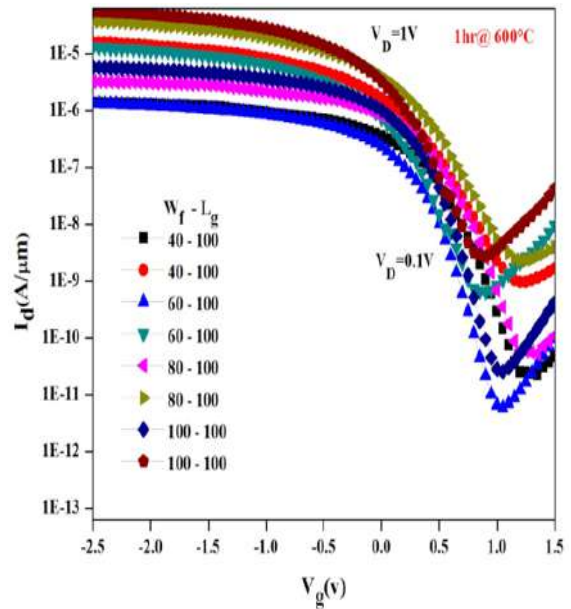


Figure 10. I_D vs V_G characteristics of the poly-Si / SiGe core-shell JAMFET at various fin width and fixed gate length of 100 nm for 1 hr at 600°C

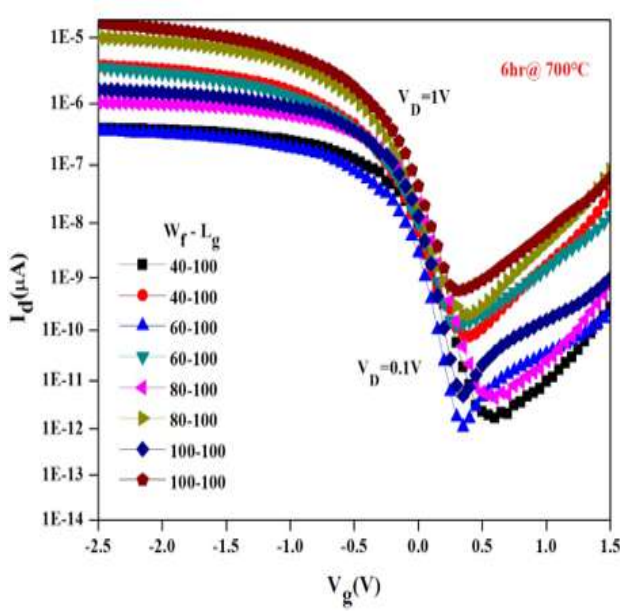


Figure 11. I_D vs V_G characteristics of the poly-Si / SiGe core-shell JAMFET at various channel width and fixed gate length of 100nm for 6hr at 700°C

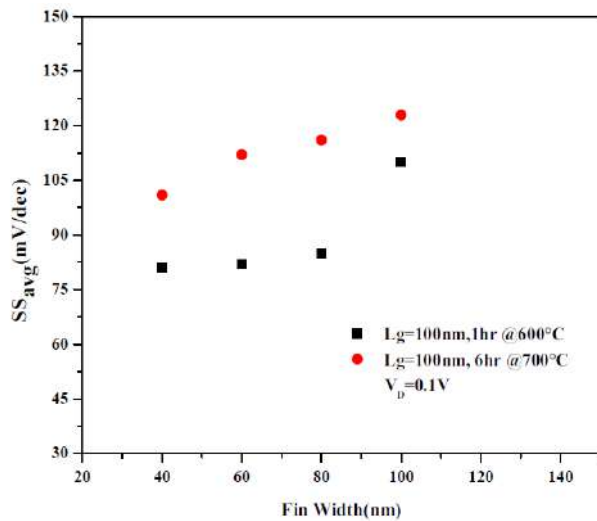


Figure 12. Distribution of subthreshold swing (SS) vs various channel width and fixed gate length of 100nm for 1hr at 600°C and 6hr at 700°C, respectively

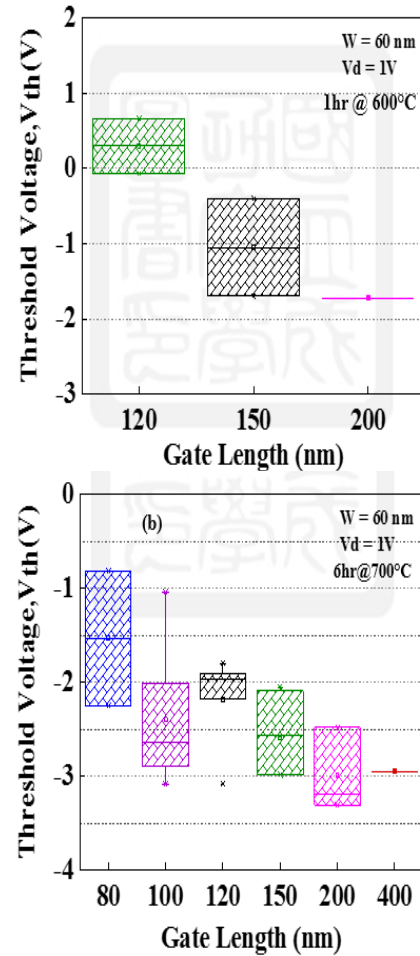


Figure 13. The comparison for statistic distributions of V_{th} for the poly-Si / SiGe core-shell JAMFETs at W_{ch} of 60 nm with different L_g for different annealing condition, (a) for 1hr at 600°C, (b) for 6hr at 700°C, respectively

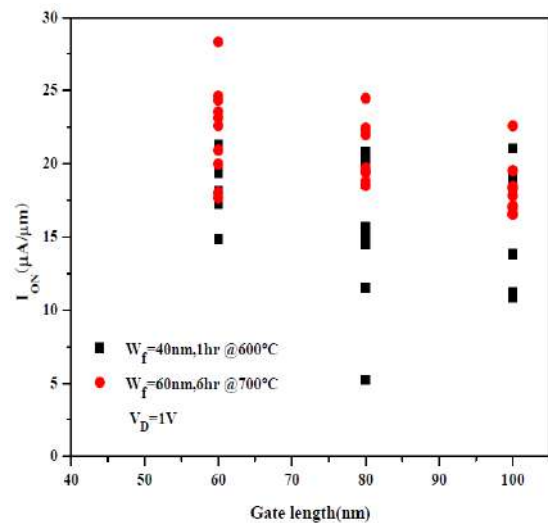


Figure 14. Dependency of ON current on gate length of poly-Si/SiGe core-shell JAMFET at channel width of 40nm for 1hr at 600°C and 60nm for 6hr at 700°C, respectively

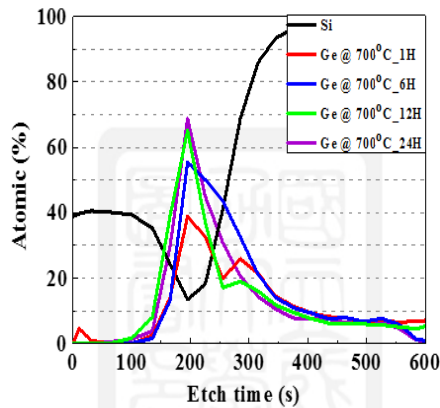


Figure 15. The depth profile of active region with four SiGe combination annealing condition, i.e. 700°C_1hr, 700°C_6hrs, 700°C_12hrs and 700°C_24hrs

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ARTICLE

Intrinsic Photoconductivity of Few-layered ZrS₂ Phototransistors via Multiterminal Measurements

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ABSTRACT

We report intrinsic photoconductivity studies on one of the least examined layered compounds, ZrS₂. Few-atomic layer ZrS₂ field-effect transistors were fabricated on the Si/SiO₂ substrate and photoconductivity measurements were performed using both two- and four-terminal configurations under the illumination of 532 nm laser source. We measured photocurrent as a function of the incident optical power at several source-drain (bias) voltages. We observe a significantly large photoconductivity when measured in the multiterminal (four-terminal) configuration compared to that in the two-terminal configuration. For an incident optical power of 90 nW, the estimated photosensitivity and the external quantum efficiency (EQE) measured in two-terminal configuration are 0.5 A/W and 120%, respectively, under a bias voltage of 650 mV. Under the same conditions, the four-terminal measurements result in much higher values for both the photoresponsivity (*R*) and EQE to 6 A/W and 1400%, respectively. This significant improvement in photoresponsivity and EQE in the four-terminal configuration may have been influenced by the reduction of contact resistance at the metal-semiconductor interface, which greatly impacts the carrier mobility of low conducting materials. This suggests that photoconductivity measurements performed through the two-terminal configuration in previous studies on ZrS₂ and other 2D materials have severely underestimated the true intrinsic properties of transition metal dichalcogenides and their remarkable potential for optoelectronic applications.

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1. Introduction

Transition Metal Dichalcogenides (TMDs) is one of the groups of two-dimensional layered crystals coupled through Van der Waals interactions that have attracted great attention in the research community for their promising potential in high performing electronic and optoelectronic devices^[1–10]. They inherit strong interaction with light as most of their bandgaps lie in the visible region, high room temperature mobilities and conductivities have given them a unique position in the semiconductor industry^[11,12]. Among the layered compounds, including those of group VIB and VIIB elements such as Mo, W and Re have been extensively studied compared to TMDs of group IVB elements; Zr, Hf and Rf. Despite the lack of attention, theoretical investigations suggest those monolayers Zr or Hf based TMDs may exhibit higher mobilities than that of group VIB counterparts^[13]. In addition, ZrX_2 compounds (X: chalcogen) predicted to show strain-induced indirect-to-direct bandgap transitions^[14,15] and even semiconductor-to-metal phase transitions^[16]. MoS_2 has been the most widely studied TMD for its electrical and optical properties, particularly due to its direct bandgap of 1.8 eV in monolayer, tunable layer dependent band structure and natural availability^[17,18]. In the monolayer, MoS_2 based field effect transistors (FETs) can achieve high ON/OFF current ratios in the order of 10^8 and high responsivity^[19,20]. Previous studies report a wide range of responsivities from mA/W to 10^4 A/W^[20–24] for monolayer and few-layered MoS_2 FETs, which depend on the incident optical power, source-drain (bias), back-gate voltages, and the type and quality of electrical contacts. Lopez-Sanchez et al.^[20] reported a high responsivity of 880 A/W under a source-drain bias voltage of 8 V and an applied gate voltage of 60 V. However, Choi et al.^[25] demonstrated that FETs based on multilayer MoS_2 have wider spectral range, high responsivity and high room temperature mobilities compared to that of monolayer MoS_2 FETs. Tsai et al.^[26] observed that few-layered MoS_2 FETs exhibit high broadband gains (13.3), high detectivities (10^{10} cmHz^{1/2}/W) and ultrafast photoresponse (rise time of 70 μ s and fall time of 110 μ s). Pak et al.^[23] reported high responsivities for monolayer MoS_2 when the FET is in the ON state ($V_g > 0$ V), however in contrast, Lee et al.^[27] observed high responsivities for few-layered MoS_2 FETs while in the OFF state ($V_g < 0$ V).

Similar to MoS_2 , other TMDs such $MoSe_2$ and WSe_2

have exhibited promising optoelectronic characteristics in both monolayer and few-layered forms^[6,8]. Abderahmane et al.^[28] observed an ultrahigh photosensitivity of 97.1 A/W and an impressive external quantum efficiency (EQE) of 22666% for a few-layered $MoSe_2$ FET at zero gate voltage. These observations signify that optoelectronic properties and transport mechanisms of MoS_2 and other TMD based FETs show a clear difference between monolayer and multilayer forms. WSe_2 is another layered TMD with bandgaps ranging from 1.3 eV (bulk) to 1.8 eV (monolayer) that shows p-type conductivity unlike MoS_2 or $MoSe_2$ ^[29]. Early studies showed that WSe_2 has a poor responsivity of 8 mW/A when the FET is in the OFF state, which increases drastically when the FET is set to the ON state, but it inherited poor switching speeds of 5 s^[30]. In contrast, our previous study on tri-layer WSe_2 crystals synthesized via chemical vapor transport (CVT) showed high speed switching behavior of 5 μ s^[6]. Our most recent study on few-layered WSe_2 FETs showed promising optoelectronic properties with high responsivity of 85 A/W and ultrahigh EQE of over 19600%^[31]. In addition, the metal contact semiconductor interface and the type of metal contacts play an important role on transport properties as they change the height of the Schottky barrier^[30]. In addition to the electrical transport, their phototransport properties can be greatly affected by contact resistance^[10,31].

Electrical transport measurements of most TMDs were conducted using two metal contacts, where both current and voltage are sourced and measured using the same terminals, in which contact resistance can dominate the transport properties. Although the contacts-driven carrier mobility can be utilized in certain applications such as in gas sensing devices^[32], it hinders the ability to investigate intrinsic properties of semiconductor devices. Some of the recent reports claim observing intrinsically higher carrier mobilities and conductivities with four terminal measurements, i.e. two terminals to measure/inject the current and the other two terminals use to sense the voltage drop across the channel^[4,9,10,33]. The advantage of four-terminal configuration is that it eliminates the influence of the contact resistance, which is vital when measuring intrinsic material transport properties of materials with low carrier mobilities. For the search of new materials and their functional properties, here we explored the phototransport properties of a few-layered ZrS_2 phototransistor with four terminals, which has not been reported in the literature. The bandgap of bulk ZrS_2 is 1.4 eV,

whereas the monolayer shows 2 eV^[34,35]. The reported mobility of few-layered ZrS₂ FET varies between 0.1 - 1 cm²/Vs^[36-38]. Further, electrical transport studies of the ZrS₂ nanobelts show extremely high photoresponsivity^[16] under UV light illumination despite its poor mobility of 1 10⁻⁵ cm²/Vs. In this study, we performed photoconductivity measurements of two-dimensional ZrS₂ FET with both two-terminal and four-terminal configurations. The main aim is to characterize intrinsic optical properties of this less known compound for various electro-optic devices as a single component or in heterostructure, which is one of the current focuses of 2D research. We observed a significant improvement in photo conductivity with the four-terminal configuration, just by restricting the role of the contact resistance. The measured photoresponsivity (*R*) and external quantum efficiency (EQE) with the four-terminal configuration was enhanced by 1200% compared to that with the two-terminal measurements for the same device. These results are in good agreement with those for WSe₂ phototransistors in our recent study^[31], in which we observed a 370% and a 461% higher photoresponsivity and EQE, respectively, with the four-terminal configuration. Thus, we believe that intrinsic electrical and optical properties of TMDs are significantly higher than their previously reported values, which opens new possibilities for improved photo-transistors and related devices where contact resistance play a significant role in measuring transport properties of the materials.

2. Results and Discussion

Few layered ZrS₂ single crystals were grown by chemical vapor transport (CVT) technique using the procedure used to grow other TMDs such as WSe₂, MoSe₂, MoS₂, ReS₂ etc.^[29,31]. Thin layers of ZrS₂ were mechanically exfoliated from bulk crystal using scotch tape technique and subsequently transferred on to a clean 270 nm thick SiO₂ film grown on a p-doped Si substrate. Single to few-layered ZrS₂ flakes were identified by optical and atomic force microscopy. These layered crystals were characterized by Raman spectroscopy to verify their composition and crystal quality. Figure 1 presents the Raman spectrum of the ZrS₂ layer using a laser source of wavelength $\lambda = 532$ nm for varying thicknesses of ZrS₂ flakes. Here, the strong signal at 330 cm⁻¹ represents the characteristic out-of-plane mode (A_{1g}) of ZrS₂, which increases with the sample thickness from bilayer (2L) to nearly twenty atomic layers (20L). The weak E_g mode is at 247 cm⁻¹. These

observed Raman modes are similar to the previously observed Raman data on few-layered ZrS₂ crystals^[35,36,39]. Figure 1 (b)-(d) display the optical micrograph images of layered ZrS₂ crystals exfoliated on to the Si/SiO₂ substrate. Raman data was collected from the several exfoliated flakes to verify the crystal quality. Supplementary Figure S1 shows AFM height image with height trace of one of the exfoliated ZrS₂ thin flakes.

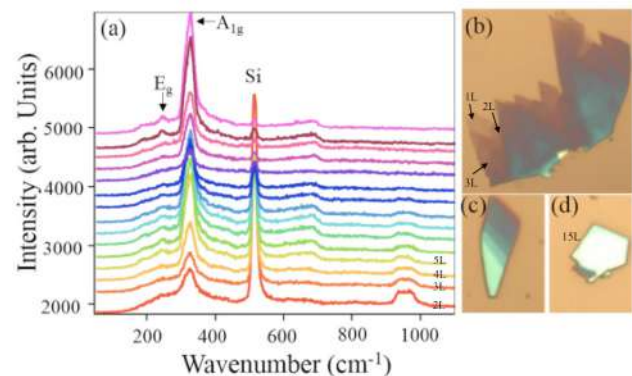


Figure 1. (a) Thickness dependent Raman spectrum of ZrS₂ crystals on Si/SiO₂ substrate. The Raman modes A_{1g} and E_g are labeled. (b)-(d) are the optical micrograph images of exfoliated ZrS₂ crystals showing single layer to few atomic layers of flakes

The field-effect transistor (FET) was fabricated on 285 nm thick *p*-doped Si/SiO₂ substrate by electron-beam lithography and electron beam evaporation technique. The devices were annealed at $T = 300^\circ\text{C}$ in forming gas followed by high vacuum annealing for 20 hours at 120°C . Figure 2(a) shows an optical micrograph image of a ZrS₂ FET device with four terminal contacts of Cr (5 nm)/Au (80 nm) with two voltage (V_1 and V_2) and two current leads (S and D) to measure the photoconductivity. To evaluate photon induced transport properties in the two-terminal configuration, the two voltage leads (S and D) were used to source voltage and measure the current of the device. The back-gate voltage (V_{bg}) was applied between the *p*-doped Si substrate and the source, and was varied to control the charge accumulation in the channel. Figure 2(b) illustrates the schematics of the FET and typical measurements obtained in the presence of a 532 nm laser under ambient conditions in a dark room environment. A dual channel Keithley Sourcemeter model 2612A was employed to apply the source-drain voltage (V_{ds}) (V_{ds} is applied between V_1 and V_2 in four-terminal configuration) and measure the source-drain current (I_{ds}), and the model 2635 was used to apply and control the gate voltage.

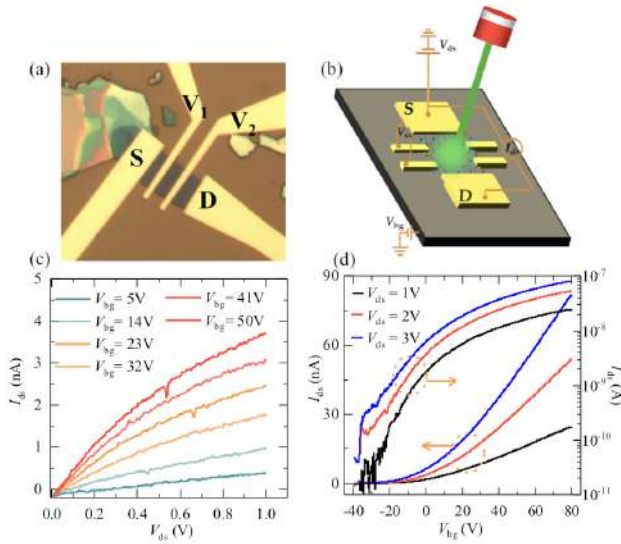


Figure 2. Optical micrograph image of ZrS₂ FET in multi-terminal contacts fabricated on the Si/SiO₂ substrate using Cr/Au (5/80 nm thick) metals. The thickness of the flake is 8-10 nm. (b) Schematic of the device and measurement scheme. (c) I_{ds} vs V_{ds} at several applied constant V_{bg} and (d) I_{ds} vs V_{bg} at constant $V_{ds} = 1, 2, 3$ V in the linear and logarithmic scale.

Figure 2(c) shows the source-drain current (I_{ds}) measured as a function of the source-drain voltage (V_{ds}) in the four-terminal configuration at several applied gate voltages (V_{bg}) from 5 V to 50 V. At lower V_{bg} values, 5 V and 14 V, the I_{ds} - V_{ds} plot shows a near linear behavior. This can be ascribed to the thermionic emission of carriers between metal contacts and ZrS₂ layer above the Schottky barrier at room temperature. However, for higher applied gate voltages (V_{bg}), the source-drain current (I_{ds}) starts displaying a non-linear variation with increasing source-drain voltage (V_{ds}). This nonlinear behavior can be attributed to non-ohmic contacts due to high Schottky barrier height between metal contacts and the few-layered ZrS₂ semiconducting channel. Figure 2(d) presents the measured I_{ds} as a function of applied gate voltage V_{bg} for constant source-drain voltages V_{ds} from 1 V to 3 V. The plot on the left current-axis displays the linear variation of I_{ds} with respect to V_{bg} . The I_{ds} with positive applied gate voltages (V_{bg}) affirms that the ZrS₂ sample is electron-doped or n-type FET. The plot using the right current-axis, shows the same variation of source-drain current (I_{ds}) in the semi-logarithmic scale as a function of V_{bg} . The observed ON to OFF current ratio of the few-layered ZrS₂ FET is 10^4 . This current is much smaller than that showed by other TMDs such as MoS₂, WSe₂ reported in the literature but agrees with the results by Zhu et al. [38] on ZrS₂ FETs. Considering the linear region ($V_{bg} > 50$ V) of the I_{ds} vs V_{bg} plot, the four-terminal field-effect mobility of the FET can be calculated by the following equation [4,31].

$$\mu = \frac{l_v}{WC_i} \frac{1}{C_i} \frac{d(I_{ds} - I_o)/V_{ds}}{dV_{bg}} \quad (1)$$

Where, l_v is the length of the channel between voltage leads V_1 and V_2 , V_{ds} ($=V_{12}$) is the voltage between V_1 and V_2 , and I_o is the off current, W is the channel width. Ratio l_v/W for our device is 0.6. C_i is the gate capacitance per unit area, given by $C_i = \epsilon\epsilon_0/d = 11.7 \times 10^{-9}$ F/cm². Where, ϵ and ϵ_0 are the dielectric constant of SiO₂ and the permittivity of free space, respectively, and d is the thickness of the dielectric material, i.e. 285 nm for the SiO₂ layer used in our experiment. The four-terminal electron-mobility of this device calculated from equation (1) is 0.5-1 cm²/Vs at room temperature. Similar mobilities of ZrS₂ FETs were reported previously on Si/SiO₂ substrate [36] or even when FET is fabricated on the h-BN substrate [37]. This value is much larger than the reported mobility by Shimazu et al. [39] measured on a 28 nm thick flake. In contrast, the four terminal mobilities of WSe₂ and MoS₂ are 145 cm²/Vs [31] and 306 cm²/Vs [4], respectively. This shows that ZrS₂ is too resistive; hence any attempt to find the two-terminal mobilities is highly affected by the contact resistance. In our previous studies on MoS₂ based FETs, we observed a similar pattern, i.e. the four-terminal mobility is significantly larger than the two-terminal mobility [4]. Mobility on ZrS₂ FET can be improved further by using suitable metal contacts or 2D metallic contacts like graphene and substrate where density of charge trap is lower than the rough Si/SiO₂ substrate. The low mobility could be attributed to defects and impurities in the crystals from CVT technique where transport agents were used for the synthesis.

We conducted both two-terminal and four-terminal photoconductivity measurements using a home-made microscope and data acquisition system equipped with a 532 nm monochromatic laser in darkroom environment. These values were compared with standard two-terminal photoconductivity measurements to evaluate the near intrinsic photoresponse of ZrS₂. More details of the experimental setup and the technique are explained in our previous study, performed to evaluate intrinsic photoresponse of WSe₂ [31]. Since the laser spot was larger than the sample size, the power illuminated on the sample P_{opt} can be estimated by,

$$P_{opt} = \frac{P}{\pi r^2} A \quad (2)$$

Where r , P and A are the radius of the laser spot, the power of the laser and the area of the sample, respectively. The radius of the laser spot (r) was measured using a lithographically patterned marker on the substrate. The dark current, I_{dark} was measured by varying the source-

drain voltage V_{ds} from 50 mV to 650 mV, in the absence of any laser illumination on the sample, i.e. $I_{\text{dark}} = I_{ds}$ for $P = 0$ W. The back-gate voltage (V_{bg}) was kept at -40 V to ensure that the device is in the OFF state. Next, I_{ds} was recorded as a function of P_{opt} by sweeping the laser power (P) from 10 nW to 50 μ W using the attenuator. The back-gate voltage (V_{bg}) was fixed at -40 V and the source-drain voltage (V_{ds}) was varied from 50 mV to 650 mV for each set of measurements. The photo-induced current (I_{ph}) was calculated by subtracting the dark current from I_{ds} obtained by illuminating with light, i.e. $I_{\text{ph}} = I_{\text{light}} - I_{\text{dark}}$, measured at the same source-drain voltage (V_{ds}). I_{ph} shows a sudden rise and then a gradual increase with P_{opt} in both two and four-terminal configurations (See supporting information Figure S2). Figure-3 (a) and (b) show the photoresponsivity (R) of the device, $R = I_{\text{ph}}/P_{\text{opt}}$, as a function of optical power (P_{opt}) illuminated on the sample, for both two-terminal and four-terminal configurations. In the logarithmic scale, R decreases almost linearly as a function of increasing optical power, indicating that trap states in ZrS_2 layer or at the interface between ZrS_2 and SiO_2 play a dominant role. This indicates that ZrS_2 based FETs are suitable for high sensitive optical detection with low optical power as the trap states can significantly alter the sensitivity and efficiency of photo detectors. In addition, both the photocurrent and the photoresponsivity gradually increase with the source-drain voltage (V_{ds}). For the two-terminal configuration, the responsivity increases from 0.03 A/W at 50 mV to 0.5 A/W at 650 mV, when the optical power on the sample (P_{opt}) is approximately 90 nW. However, in the four-terminal configuration, the responsivity rises from 0.14 A/W at 50 mV to 6 A/W at 650 mV for the same (P_{opt}). Thus, the responsivity values extracted from the four-terminal measurements are significantly larger than the values extracted from two-terminal measurements. This increase in responsivity in four-terminal measurements is due to the elimination of contact resistance associated with the metal and semiconductor junction. The highest responsivity, R for two-terminal measurements, 0.5 A/W at $V_{ds} = 650$ mV and $V_{bg} = -40$ V, jumps to 6 A/W in the four-terminal measurements under the same V_{ds} and V_{bg} , which is nearly a 1200% enhancement. Our measured R value is much higher than the reported value for the same material by Wang et al. [36]. These values of R are also two orders of magnitude higher than the reported results by Mattinen et al., where they used Atomic Layer Deposition (ALD) method to synthesize large scale ZrS_2 crystals [40]. By fitting the responsivity as a function of optical power on the sample to the power law $R \propto P_{\text{opt}}^{-\gamma}$, when $V_{ds} = 650$ mV, we obtained the $\gamma = 0.84$ and $\gamma = 0.87$ for the two-terminal and four-terminal configurations, respec-

tively.

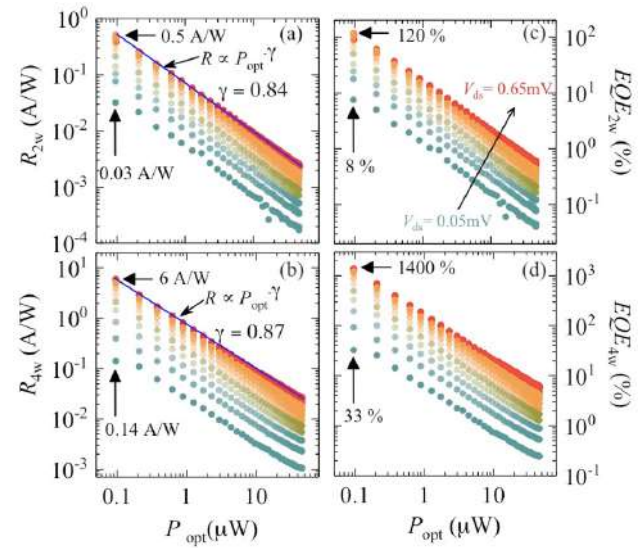


Figure 3. (a) and (b) display the photoresponsivity measured in two- and four-terminal configurations respectively as a function of incident optical power. (c) and (d) are the estimated external quantum efficiencies (EQE) for two- and four-terminal measurements for few layered ZrS_2 phototransistor. Blue solid lines in (a) and (b) are the least square fit to the experimental data

Next, we estimate the external quantum efficiency (EQE), which is defined as the number of electron-hole pairs generated by the number of photons illuminated on the phototransistor, using the following expression [10,31].

$$EQE = \frac{I_{\text{ph}}}{P_{\text{opt}}} \frac{hc}{\lambda q} = R \times \frac{hc}{\lambda q} \quad (3)$$

Where, P_{opt} is the optical power incident on the sample, h is the plank constant, c is the speed of light, λ is the wavelength of the laser source, and q is the electron charge. As shown in Figure 3 (c) and (d), the EQE follow a similar variation as R as a function of optical power ($EQE \propto R^{-\gamma}$). In the logarithmic scale, EQE decreases linearly with increasing P_{opt} at constant source-drain and back-gate voltages in the FET's OFF state, and EQE increases with the source-drain voltage for a given optical power on the sample. EQE increases from 8% to 120% when the source-drain voltage is increased from 50 mV to 650 mV in two-terminal measurements, however, it jumps from 33% to 1400% for the same source-drain voltage interval in the four-terminal configuration. This very high quantum efficiency of 1400% in four-terminal measurements compared to that estimated through two-terminal measurements, 120%, signifies that the four-terminal configuration produces a considerably higher EQE. This is nearly 1200% increase compared to the much adopted two-terminal configuration. In one of our previous studies to

determine the photoresponsivity of WSe₂, we made a similar observation, where the EQE increased by 344% when it was estimated using a four-terminal configuration^[31]. The linear decrease of EQE with increasing incident optical power has been observed for other 2D crystals^[10,31] and believed to be a result of electron-hole recombination, which may have been stimulated by the radiation. However, the observed high EQE values in this study indicate that the recombination process is either hindered or enhanced by the generation of electron-hole pairs caused by the large surface to volume ratio of our few-layered ZrS₂ device. EQE values exceeding 100% suggests that one photon may have generated multiple electron-hole pairs, however the excitation frequency remains too low for this to occur. Studies performed by Li et. al.^[41] and Ulanganathan et. al.^[42] claim that high EQE and photoresponsivity are due to localized trapped states that slows the recombination process. They argue that one type of photo generated carriers is trapped in localized states, which expands the lifespan of the other carriers to circulate multiple times through the channel and source meters before the recombination. This process enhances the effective gain of the device as one photon seems to have created a substantially large photo-induced current due to slow a recombination process. This process yields EQE values exceeding 100%.

This study was performed for a single wavelength ($\lambda=532$ nm) as we focused on finding the dependence of photoresponsivity and photoinduced current on incident optical power in a multi-terminal configuration. The dark current was primarily due to thermally excited minority charge carriers. When the device is illuminated, the total current comes from thermally excited minority charge carriers (dark current), photogenerated current by electron-hole pairs, and the current induced by the photo thermoelectric effect (PTE) at the metal-semiconductor interface^[43], as the whole device is exposed to the laser radiation, i.e. both the Cr/Au metal contacts and ZrS₂ semiconducting channel. Under laser illumination, the metal contacts become warmer than the semiconductor and their difference in Seebeck coefficients induces a net current flow across the metal-semiconductor interface, from the metal (hot) to semiconductor (cold)^[43]. Since the size of the laser spot (700-800 μm^2) is much larger than the area of the device including metal contacts (100 μm^2), the net current flow across the channel due to PTE should be zero, as the currents from the two metal contacts to the semiconductor flow on opposite directions and cancels out. This leaves the dark current and the photogenerated current as contributing factors when the device is under illumination.

The dependence of R and EQE in the four-terminal configuration with the source-drain voltage (V_{ds}) in the logarithmic scale is shown in Figure 4, based on extracted data from Figure 3 (c) and (d), for selected incident optical

powers. Upon fitting with the power law ($R \propto V^{-\alpha}$), we found that the dependence of R with V_{ds} is slightly non-linear with exponent $\alpha=1.6$. The highest value estimated for R is 6 A/W at $V_{ds} = 650$ mV and $P_{\text{opt}} = \mu\text{W}$ that significantly decreases as we increase the optical power. However, previous studies have reported very high responsivities for WSe₂ phototransistors under ultra-low incident optical power and higher bias voltages^[28]. This mismatch might have come from the low absorption of ZrS₂ at 532 nm compared to that of WSe₂. ZrS₂ shows high absorption in the range of 300 nm to 400 nm in heterostructure geometry with *h*-BN or graphene compared to 532 nm in WSe₂^[44]. The responsivity also largely depends upon the incident optical power, applied drain-source and gate voltages used. As an example, a high R of 880 A/W was reported for monolayer MoS₂ based FETs under a very low incident optical power of 150 pW ($V_{ds} = 8$ V and $V_{bg} = -70$ V), which sharply dropped to 4 A/W as the incident optical power increased to 250 nW^[20]. In comparison, the R value estimated in our study for ZrS₂ FET at 250 nW (0.25 μW) and $V_{ds} = 650$ mV (0.65 V) is approximately 2 A/W (Figure 3 b), which can be extrapolated to approximately 110 A/W at $V_{ds} = 8$ V (Figure 4 a). This is in good agreement with our previous study on few-layered WSe₂, which shows a very high R value when it was measured with the four-terminal configuration ($R = 85$ A/W at $P_{\text{opt}} = 248$ nW and $V_{ds} = 1$ V)^[31]. This illustrates that the four-terminal configuration-based measurements have resulted in a much higher R and EQE values than those of previously reported on ZrS₂ phototransistor.

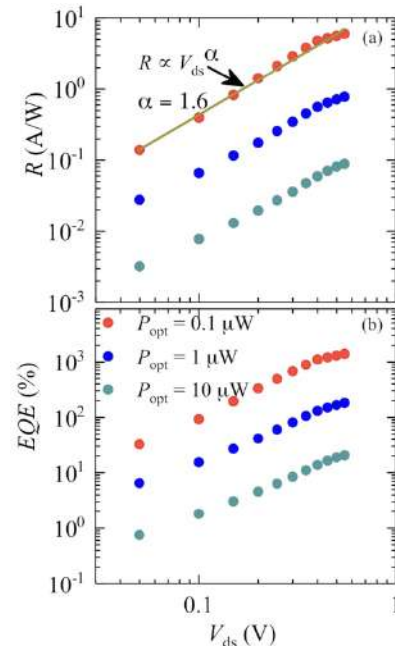


Figure 4. (a) Responsivity and (b) EQE as a function of V_{ds} extracted at constant illuminated optical power. Red line represents the fitting of R in power law to extract the exponent α

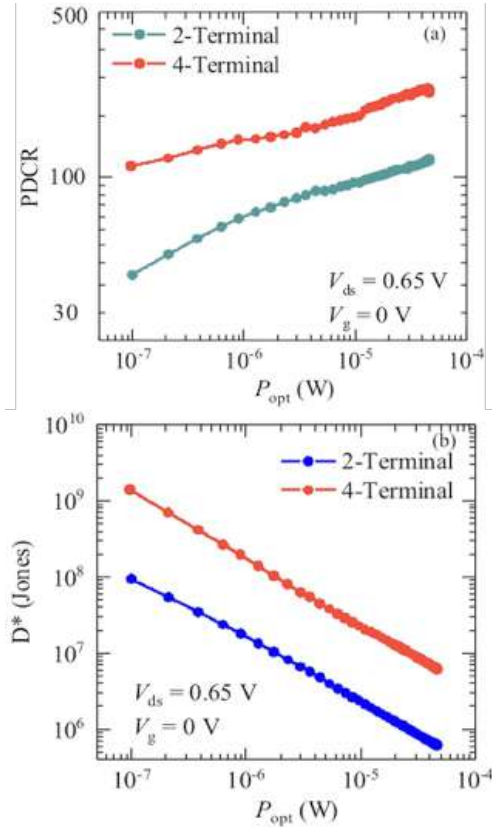


Figure 5. (a) PDCR and (b) Detectivity of few layered ZrS₂ phototransistor as a function of illuminated optical power

We further explored the photo dark current ratio (PDCR) and detectivity (D^*) of our ZrS₂ based photo-transistor as presented in Figure 5, which demonstrates the photodetection application and the figure of merit to use for the performance of photodetector of ZrS₂ based phototransistor. PDCR is estimated by measuring the ratio of photocurrent and dark current ($PDCR = (I_{light} - I_{dark})/I_{dark} = I_{ph}/I_{dark}$, where I_{light} is the current measured with light illumination and I_{dark} is the dark current with no light illumination. Figure 5 (a) shows the PDCR values of few layered ZrS₂ measured in two and four-terminal configurations as a function of increasing optical power. The PDCR values measured in 4-terminal method ranges between 100 at 0.1 μ W and 275 at 0.4 mW optical power under applied $V_{ds} = 0.65$ V, which are much higher than some of the reported photodetectors currently in use such as AlN, GaN, SiC, Ga₂O₃ etc. [45-48] and similar to the MoS₂ photodetector reported earlier [26]. The specific detectivity is extracted from the relation given by $D^* = R\sqrt{A}/\sqrt{2qI_{dark}}$, where R is the photoresponsivity, A is the area of the detector, q is the unit of charge, and I_{dark} is the dark current. As shown in Figure 5 b, D^* varies linearly with optical power from 10^7 Jones at 1 mW power to 10^9 Jones at 0.1 μ W incident

optical power in the OFF state of the transistor ($V_{bg} = 0$ V) and small $V_{ds} = 0.065$ V. Similar detectivities were also reported on few layered MoS₂ [26]. Higher value of the detectivity can be obtained in the ON state of the transistor and by applying higher V_{ds} [30].

3. Conclusion

We report the intrinsic photo transport properties of few layered ZrS₂ phototransistors measured with multi-terminal configuration. We studied the dependence of photocurrent, photoresponsivity and the external quantum efficiency as a function of incident optical power and bias voltage when the FET is in the OFF state at a fixed back-gate voltage. The estimated photoresponsivity R and EQE values show that four-terminal measurements result extremely high values, at least one order of magnitude higher than that with two-terminal configurations. This clearly indicates that the optical properties of FETs based on few-layered TMDs can be significantly enhanced by minimizing the contact resistance. Here, the ZrS₂ FET with approximately 10 atomic layers of ZrS₂ shows an impressive R and EQE values through four-terminal measurements representing an improvement of 1200%, over the values estimated through traditional two-terminal configuration. Furthermore, few-layered ZrS₂ shows an excellent photo sensitivity factor (PDCR) that is up to 275 at low applied bias and zero gate voltage when measured in four-terminal configuration. Few-layered ZrS₂ also shows high detectivity at low incident optical power. This signifies that a four-terminal configuration is more suitable when investigating intrinsic optoelectronic properties of TMD based FETs, particularly 2D materials where contact resistance plays a crucial role to determinate of incident the intrinsic transport properties, which can be ultimately utilized for the fabrications of devices that require high yields and high sensitivities. We believe that measuring the intrinsic optoelectronic properties of 2D materials, TMDs would make them promising candidates for a wide range of future applications. The electrical and optical properties of ZrS₂ reported here will guide to fabricate heterostructure devices based 2D TMDS where ZrS₂ can provide the high yields and sensitivities required for optical applications.

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Supporting Information

The supplementary materials contain the Figure S1, which is the variation of photocurrent as a function of incident optical power under different bias voltages for both two-terminal and four-terminal configurations.

Supplementary Materials

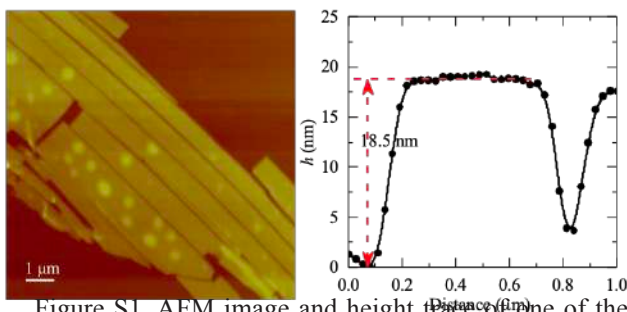


Figure S1. AFM image and height trace of one of the exfoliated flakes of ZrS_2 on to Si/SiO_2 substrate

Note: AFM height measurements were performed on several exfoliated crystals of ZrS_2 on Si/SiO_2 substrate using Veeco Dimension 3100 AFM setup. The flakes used for optical measurements are from 8 nm to 15 nm thick.

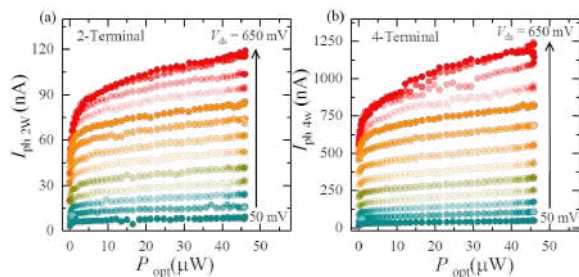


Figure S2. Photocurrent vs incident optical power

Note: The photocurrent as a function of incident optical power under several bias voltages for (a) two-terminal configuration (b) four-terminal configuration ($I_{\text{ph}2\text{W}}$ and $I_{\text{ph}4\text{W}}$ denote the photocurrent with two and four-terminal configurations, respectively). The four-terminal photocurrent is significantly larger ($>1000\%$) than the two-terminal current. Under both configurations, the photocurrent increases with the bias voltage.

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ARTICLE

Infrastructure of Synchrotronic Biosensor Based on Semiconductor Device Fabrication for Tracking, Monitoring, Imaging, Measuring, Diagnosing and Detecting Cancer Cells

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Measuring

Diagnosing

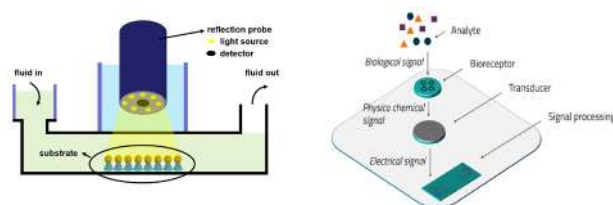
Detecting

Cancer Cells

Tris(2,2'-bipyridyl) ruthenium(II)(Ru(bpy)₃²⁺)

ABSTRACT

Copper Zinc Antimony Sulfide (CZAS) is derived from Copper Antimony Sulfide (CAS), a famatinite class of compound. In the current paper, the first step for using Copper, Zinc, Antimony and Sulfide as materials in manufacturing synchrotronic biosensor-namely increasing the sensitivity of biosensor through creating Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor and using it instead of Copper Tin Sulfide, CTS (Cu₂SnS₃) for tracking, monitoring, imaging, measuring, diagnosing and detecting cancer cells, is evaluated. Further, optimization of tris(2,2'-bipyridyl)ruthenium(II)(Ru(bpy)₃²⁺) concentrations and Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor as two main and effective materials in the intensity of synchrotron for tracking, monitoring, imaging, measuring, diagnosing and detecting cancer cells are considered so that the highest sensitivity obtains. In this regard, various concentrations of two materials were prepared and photon emission was investigated in the absence of cancer cells. On the other hand, cancer diagnosis requires the analysis of images and attributes as well as collecting many clinical and mammography variables. In diagnosis of cancer, it is important to determine whether a tumor is benign or malignant. The information about cancer risk prediction along with the type of tumor are crucial for patients and effective medical decision making. An ideal diagnostic system could effectively distinguish between benign and malignant cells; however, such a system has not been created yet. In this study, a model is developed to improve the prediction probability of cancer. It is necessary to have such a prediction model as the survival probability of cancer is high when patients are diagnosed at early stages.



Schematic of infrastructure of synchrotronic biosensor based on semiconductor device fabrication for tracking, monitoring, imaging, measuring, diagnosing and detecting cancer cells

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1. Introduction

Biosensors are systems for tracking, monitoring, imaging, measuring, diagnosing and detecting the concentration of cancer cells such as proteins, enzymes, nuclides and etc. which produce by various methods and materials depending on the type of biosensor and cancer cells. In optical method of synchrotron, a synchrotron excites at the presence of activator agent due to applying electrical potential and hence, emits photon. In optical synchrotron biosensor, the concentration of cancer cells can be measured using this method and stabilizing the synchrotron radiation on the cancer cells. In other words, cancer cells play the role of electrical potential carrier to synchrotron radiation. Hence, the applied potential to synchrotron radiation varies with concentration of cancer cells and therefore, the intensity of emitted photons varies^[1-47]. The advantages of synchrotron method compared to other optical methods are (a) It does not necessary to have an excitation source which cause to reduction of optical interferences; (b) Having strong time and position separation power; (c) Simplicity, low cost, high speed and low time of measurement^[48-92].

In the produced optical biosensor, as the first sample in the country, Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor was used which is one of the most used synchrotron radiation, applied in manufacture of synchrotron biosensors due to its high quantum efficiency and small size. Small size of Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor leads to its easy conjugation with cancer cells which minimizes the interference in immune system of cancer cells^[93-121]. In the produced optical sensor, Tris(2,2'-bipyridyl)ruthenium(II) (Ru(bpy)₃2+) is used as activator agent for Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor.

One of the basic characteristics of biosensor is its high sensitivity. Sensitivity of a biosensor is the minimum amount of concentration detection of cancer cells. According to this definition, sensitivity of the produced biosensor increases proportional to increase in intensity of emitted photons from synchrotron radiation. Hence, Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor was used for this reason.

Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor nanoparticles enhance the intensity of photons due to some advantages. Two time-ionized CZAS nanoparticles easily coop Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor ions due to having negative charge and

enhance the optical stability of Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor because of their optical property. At the other hand, as these molecules are of large active surface, they are able to charge (coop) a large amount of Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor molecules. However, CZAS nanoparticles cannot individually stabilize on cancer cells such as antibodies and hence, Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor nanoparticles are used to solve this problem^[122-184].

The produced Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor nanoparticles have negative charge on their surfaces due to the manufacture type and therefore, they can easily absorb functional groups with positive charge (such as amino groups). Many cancer cells are of functional groups with positive charge. To settle Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor nanoparticles with negative charge on CZAS, layers with positive charge such as amino groups can be used. Due to small size of nanoparticles, a large number of them settle on CZAS (Figure 1). In addition, regarding the fact that Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor nanoparticles are strong electric conductors, they enhance electron transferring process (electrical potential) to Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor coop into CZAS^[185-217].

In this project we applied several machine learning techniques on the Wisconsin Diagnostic Breast Cancer data set to classify the cancer based on the feature extracted from images as benign or malignant^[50-73].

In the current age, pancreatic cancer is one of the worst forms of cancer. The complications of pancreatic include five types of pancreatitis, benign tumors, malignant tumors, benign cysts and malignant cysts^[1-27]. This cancer has a few clinical symptoms than other cancers. Also, if not treated in a timely manner, it also causes other organs of the body and the patient chance of survival is greatly reduced. One of the ways to detect this disease is to use CT scan images. But the appearance of pancreatic complications is very different in a similar category, and their tissue is very similar to healthy abdominal tissues^[28-49]. For this reason, it's very difficult to identify the range of complications. In this study, the data contained 151CT scan images. These images are divided into five classes of pancreatitis, malignant tumors, benign tumors, malignant cysts, benign cysts and a healthy class. The pancreatic complications are varied and different, if the diagnostic system is based on simple experts; the possibility of

achieving high detection accuracy is not possible. According to the results of this study, lonely no classification can detect all diseases and combining these methods is the best option. Therefore, in this study we have achieved high accuracy in prediction (690. 69) by combining the perception, convolution and SVM neural networks.

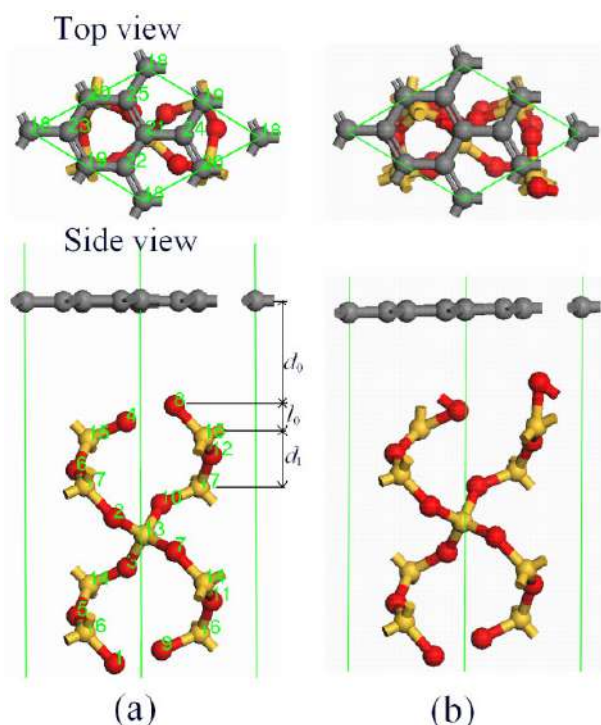


Figure 1. Schematic view of Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor

In the current experimental work, in addition to sample preparation and manufacturing sensor device, the effect of semiconductor concentration also is investigated. As it is necessary to prevent any interference on the structure of Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor, this issue is investigated in sample preparation and using them in electrochemical system^[218-358].

2. Materials, Methods and Techniques

Polyurethane are biocompatible compounds with variety applications in the biomedical fields mostly as drug delivery vehicles. Their various applications are due to their Maneuverable structure with different blocks of diols and isocyanides. In the new presented work, magnetic polyurethane was used as drug carrier which formed of the reaction of Poly-caprolacton and isophoren diisocyanate and finally cyclodextrin as the cross linker. Characterization of the final polymer and certainty of its formation was done through different analytical methods such as FT-IR, TGA, XRD, SEM, TEM and VSM. On the other hand, the

percentage of the magnetic nanoparticles in the polymer matrices was tracked using thermal gravimetry analysis. This Nano drug carrier was used for in vitro delivering pharmaceutical agent of doxorubicin. The amount of drug loading and percentage and manner of the drug release were investigated using concentration profile. Cytotoxicity of Nano drug carrier was evaluated using calorimetric method called methylthiazolotetrazolium (MTT) assay on the MCF-7 cell lines and according to the results presented system is very profitable and proper one for delivering Doxorubicin anti-cancer drug^[74-93].

Melittin (MEL) is a kind of catalytic peptide that isolated from bee venom. Catalytic peptides are promising drugs for cancer treatment because cancer cells are less likely to develop resistance to a membrane-perturbing agent. However, their nonspecific cytotoxicity has limited their therapeutic applications. In this study, we use citric acid stabilized Fe_3O_4 magnetic nanoparticles (CA-MNP) as potential magnetic carriers for target delivery of melittin to tumor sites. The morphology and surface functionalization of these magnetic Nano carriers were studied by field emission scanning electron microscopy (FESEM) and Fourier transform infrared. The loading and release profile of MEL were studied by UV spectrophotometry. The results indicate that these magnetic Nano carriers have the high drug loading efficiency and the pH-dependent release behavior. The in vitro cytotoxicity of the MEL-loaded CA-MNP on the MCF-7 breast cancer cell line is similar to that of free MEL in solution at equivalent doses^[94-104].

The interest in exploring more effective methods for cancer treatment has increased widely in recent years. In clinical studies it is difficult to determine the temperature distribution in both normal tissue and in tumor during hyperthermia treatment since temperature can be measured in limited number of positions in tissue or tumor. Simulation studies can play crucial role in physician's perception of the temperature distribution in tissue. Hyperthermia treatment is facing some unsolved problems such as the appropriate dosage of magnetic Nano particles required to achieve the optimum temperature which results in apoptosis in tumor cells. In this study, a 2D computational model is created in COMSOL Metaphysics in order to analyze temperature distribution in both normal tissue and tumor during hyperthermia treatment using various dosages of magnetic Nano particles. Temperature distribution is achieved by considering various layers from wave source through to the tumor and also by taking into account the amount of heat generated through the Brownian rotation and the Neel relaxation. Simulations of a spherical tumor located in ellipse tissue were designed. A systematical

variation in dosage has been performed. Temperature distribution and maximum temperature in steady state and effect of the dosage of Nano particles^[105-117].

In this study, cobalt Ferrite nanoparticles with inverse spinel structures were obtained using co-precipitation of cobalt and iron nitrates. Ammonia 15% was used as an alkaline agent for pH adjustment. Besides, we used oleic acid to coat the cobalt ferrite nanoparticles. XRD analysis showed that the samples included spinel ferrite structure. According to the results of SEM the distribution of the particles was homogeneous and the particles were uniform, and pseudo-spherical in shape. The magnetic properties of the material were analyzed by the VSM that showed the relationship between super-paramagnetic properties of the material and particle size. In this research, for the first time, anti-cancer effects of cobalt ferrite nanoparticles on K562 cell line as an experimental model of acute myeloid leukemia (CML) were examined. Because this compound has the potential to induce differentiation and apoptosis, it can be used in conjunction with other pharmaceutical compounds as a promising candidate for the treatment of blood cancer patients^[118-143].

Cancer, as a leading contributor to the global disease burden is characterized by the uncontrolled growth of cells in the body, which makes it one of the most difficult and complex diseases to treat. Dietary sources of natural products including fruits and vegetables have been reported to be associated with reduced risk of a variety of tumors and to have anti-cancer benefits, apart from being a good source of nutrients. Thus, among major groups of anti-cancer drugs, plant extracts have received considerable attention to discover promising cancer therapeutic agents from natural sources. Great interest is currently centered on the biologic activities of quercetin a polyphenol belonging to the class of flavonoids, natural products well known for their beneficial effects on health, long before their biochemical characterization. onion skin waste is rich in bioactive compounds such as phenolic and flavonoids. In this direction, Quercetin, a natural compound abundantly present in Onion skin has great therapeutic potential in the prevention and treatment of cancer. This review focuses on anti-cancer potential of Quercetin with current advancements for its implementation in treatment of cancers^[144-173].

Breast cancer is the uncontrolled growth of abnormal cells in the breast area and it is one of the widespread causes of mortality in today's world. So that 8000 people are diagnosed with breast cancer of a year in the world. The exact and precise diagnosis is considered as the vital point in the process of treatment. Among the various methods of screening, thermography is a non-invasive

and safe method to detect breast cancer. In this work, a classification algorithm of thermograms with the purpose of detection of breast cancer from gray level co-occurrence matrix based features texture has been proposed. For this purpose, 52 images from the breast of healthy and unhealthy people from the data were collected. The preprocessing and segmentation of data was performed in gray level for the creation of temperature matrix. Finally, the gray level co-occurrence matrix based features was extracted from the matrix and the collection of features using Manhattan technique was the input for weighted K-nearest neighbor classifier. The result of Accuracy was 85.6, Sensitivity was 91.7 and Specificity Index was 81.2 selected as the optimal structure compared to other methods that have been proposed so far^[190-203].

Cancer is the third leading cause of death in the world, as well as breast cancer is the second most common cause of death among women in the world. According to calculations by the National Cancer Institute of the United States, one person of every eight women will be diagnosed with breast cancer. Unfortunately, the age of cancer in the world is a decade younger than other developed countries. Therefore, early diagnosis of this disease is essential in the healing process. With detect and remove cancerous tumors in the early stages before spreading to neighboring areas, cancer threats be stopped. Among the various methods of screening, thermography is a non-invasive and safe method to detect breast cancer. In research, at first paid to the automatically way that in this regard, Kenny edge and Hough transform have been enjoying and then a thermography classification algorithm to detect breast cancer based on certain characteristics extraction of the tissue in gray level co-occurrence matrix is provided. For this purpose, 68 healthy and unhealthy images of the breast are collected from the database. Finally, the features set as input are given into the support vector machine classifier. The result of Accuracy was 87.3, Sensitivity was 89.6 and Specificity Index was 83.9 selected as the optimal structure compared to other methods that have been proposed so far^[204-224].

Cancer caused by cells goes out of correct pathways. This cell can invade to surrounding healthy cells. There are over 100 different types of cancer and all of them classified by the type of cell that affected. Usually malignancy of gastric cancer starting from layer of the stomach. Gastric cancer has been mentioned as a third cause of death in the world. According to the statistical results, we can see the high frequency of gastric cancer in, Japan, China, Central and South America, Eastern Europe and parts of the middle east. Higher rates usually have been seen group with lower socioeconomic^[225-241]. Some signs of this

cancer are indigestion or heartburn, vomiting, diarrhea, constipation and having blood in stool. Stomach cancer usually detects in early stage. Each factor that increase the chance of developing cancer is known as a risk factor. Some factors that may increase the risk of stomach cancer are: Age, gender, bacteria, family history, race, diet, previous surgery, smoke and obesity^[242-266]. Diagnosis of gastric cancer at first are obtained from laboratory tests and biopsy of stomach with endoscopy. In the next step, cancer may be treated with Surgery, radiation therapy, chemotherapy or immunotherapy^[267-284].

Lung cancer is one of the deadliest cancers, such that it causes more deaths compared to breast cancer, colon cancer and prostate cancer and it is mainly because it cannot be diagnosed at early stages due to shortage of symptoms, such that survival rate of patients for 5 years after surgery is only 14%; while diagnosing the disease at early stages increases this probability to 70%. Increasing growth of this disease, difficulty of its diagnosis from images and importance of diagnosis at early stages requires CAD methods with high accuracy. In order to realize this important, a novel algorithm is proposed in this study which selects features online using genetic algorithm and statistical functions. Our purpose is to separate effective features among available features. In order to classify data, a series of data called feature is required for which disease features are used. In many datasets, some features do not affect decisions and they are additional. So selecting an appropriate subset of inputs can be effective in classification accuracy and its speed. For this purpose, genetic algorithm with an objective function based on data sparsity and statistical concepts. The proposed method is implemented and results indicate high accuracy of this algorithm in selecting effective features and increasing accuracy of the classifier compared to basic methods and other studies^[285-299].

Cancer is a major cause of death with more than 10 million annual patients. It is possible that this number reaches 15 million patients per year by 2020. Though chemotherapy has largely been successful in controlling and treating cancer, live tissue damage, systemic toxicity and side effects in this method are among the issues that cannot be overlooked. In order to reduce the negative effects of anticancer drugs on normal tissues, we need to design Nano-sized carriers that can pass the safety barriers and body tissues and reach their target site. In this work, the size and zeta potential of Nano-carriers PLGA-Cs-Paclitaxel were evaluated. Chitosan connection in physical or conjugated forms may lead to a significant increase of polydispersity. According to the study carried out on the concentration of Chitosan and the type of absorption,

it was concluded that nanoparticles size increases with higher concentrations of Chitosan. The zeta potential will increase, provided the conjugation of Chitosan is higher than physical adsorption^[300-311].

Cancer stem cells (CSCs) are rare sub-population of tumor with ability to differentiate and self-renew. Some properties of CSCs such as increased ability to repair damaged DNA/RNA, as well as increased expression of transporters responsible for drug efflux make them main agents for resistance to chemotherapy. In colon cancer, FOLFOX is a common therapy. In this study, we have analyzed the effects of FOLFOX on CSCs population of colon cancer cell line. Results show that in addition to a dose-dependent reduction in cell viability, FOLFOX caused a decrease in SP cells relative to untreated controls^[344-358].

For the detection of DNA/RNA hybridization, a new electrochemical biosensor was developed on the basis of the interaction of Doxorubicine (DOX) with 22-mer oligonucleotides (from human cancer) a simple bio sensing design to yield an ultrasensitive electrochemical biosensor for cancer biomarker detection on Screen Printed Gold Electrodes (SPGE) without use of any modification on electrode surface perhaps direct detection with the help of electroactive label (DOX) and MicroRNA92a (miRNA) as an biomarker selected for being up-regulated in cancer. The biosensor was assembled in two stages the immobilization of the probe that was modified on an SPGE and second stage of target hybridization of completely match strand electroactive label DOX has been used after hybridization process which is an intercalator with our miRNA strands as a redox indicator for amplifying the electrochemical signal of miRNA 92a. For conformation electrochemical techniques including Cyclic Voltammetry (CV) and Differential Pulse Voltammetry (DPV). were used and hybridization was observed successfully. The final biosensor provided a sensitive detection of miRNA 92a with good selectivity.

Based on the researches, one of the most common cancer among the men is malignant cancer. Which seen after surgery and gland removing completely the amount of PSA in patient increases again and available drugs which have severe side effects cannot effect on rising the PSA. One parameter that without it cancer cells are not able to reproduce is Glutamine Amino Acid. With studying humanity biochemical pathway and Glutamine Amino Acid reabsorbing pathways by cancer cells we understand that two material Ursolic Acid and Resveratrol could dock with a lots of Allosteric Enzymes inside the reabsorbing pathway Glutamine Amino Acid by cancer cells. That inactive enzymes therefore more than 90 % reabsorbing

pathways Glutamine Amino Acid will be closed. Docking these two materials Ursolic Acid and Resveratrol with Allosteric enzymes reabsorbing pathways Glutamine Amino Acid by cancer cells and inactivating enzymes with software Autodock-vina and QSAR has been checked. Also Curcumin could stimulate Apoptosis in cancer cells. Since three above substances (Resveratrol, Curcumin, Ursolic acid) exist in available compounds like skin of red apple, turmeric and black grapes, men above forty years old can reduce risk of cancer by combining a big apple with some turmeric and black grapes (as a potion or juice). So it can protect from cancer. After prostate surgery, PSA may raise again. Consumption of this potion in these cases can replace current medications with several adverse effects. Since we could find Ursolic Acid in red apple skin and Resveratrol in black grape and Curcumin in Turmeric, we could extraction and combine these three material and with determining the amount of doze make a medicine. Then doing the next steps like animal test, toxic test and human test. Base on human gene plan (HGP) and humanity biochemical pathway lack of Allosteric enzymes which by two material Ursolic Acid and Resveratrol will be inactivate and therefore non-proliferation cancer cells.

Chemotherapy resistance of cancers have become a big challenge in modern medicine. Recently, in order to overcome the drug resistance issue, producing novel drug which used with previous ones as multidrug treatment became an alternative. One of the compounds that have drawn much attention in this regard is chromenes. Chromenes have a heterocyclic structure with gamma benzopyrone, and anti-cancer activities. Studies tried to produce new derivative of chromenes which have better effect on cancer therapy. In this investigation we produced four novel derivatives of chromenes and studied the effect of these compounds on the human acute lymphoblastic leukemia cell line of MOLT4. A series of novel 4-hydroxycoumarin has been synthesized via multi-step protocol. The structure of the new compound was established using spectroscopic method (H-NMR, C-NMR). MOLT4 cells were cultured in RPMI medium with 10% fetal bovine serum. The cytotoxic effect of different concentrations (0, 50, 250, 500 and 1000 nM) of novel synthetic compounds were evaluated by the MTT assay and cell counting after different incubation times (24, 48, 72 h). These compounds decreased viability of the MOLT4 cells in a time- and dose-dependent manner. Notably, meaningful differences were found between all concentrations and control groups. However, C2 had fewer IC50 in comparison to other ones. Interestingly, this derivative showed significantly cell toxicity at the concentration of Nano-Molar, while previously reported ones have cell toxicity at mi-

cro-Molar concentrations. Dihydrochromeno (3, 4-b) chromenes have anti-neoplastic effects on MOLT4 by inducing of apoptosis. Further studies are needed to find exact mechanisms of its effect.

3. Results and Discussion

In order to recognize the size of produced Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor nanoparticles, SEM imaging was used. Figure (2) shows a sample of SEM image produced from Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor nanoparticles. In this regard, the average size of these particles is between 15-20 (nm).

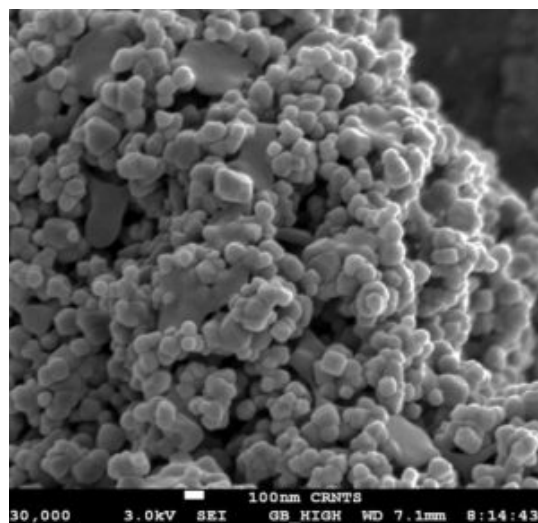


Figure 2. SEM image for the produced Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor nanoparticles

Figures 3a and 3b show SEM images for Cu-Zn and Cu-Zn-Sb, respectively. Size of these nanoparticles is about 50 (nm). By comparing the obtained sizes, it was indicated that Cu-Zn nanoparticles are able to load a large number of Cu-Zn-Sb nanoparticles.

Figure 4 shows attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectra of Cu, Cu-Zn and Cu-Zn-Sb semiconductor. Comparison of absorptive curves indicate 5 (nm) shift of wavelength in the spectrum of Cu-Zn at 450 (nm) which confirms coupling of Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor into Cu-Zn-Sb. In addition, according to emission curve, semiconductor manufacture does not change the emitted spectrum of Cu and its synchrotron nature and increasing the emission intensity of semiconductor indicates coupling a large number of Copper Zinc Antimony Sulfide, CZAS ($\text{Cu}_{1.18}\text{Zn}_{0.40}\text{Sb}_{1.90}\text{S}_{7.2}$) semiconductor molecules.

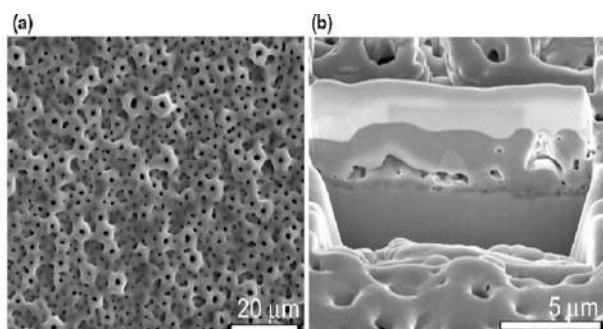


Figure 3. SEM images for (a) Cu-Zn and (b) Cu-Zn-Sb, respectively

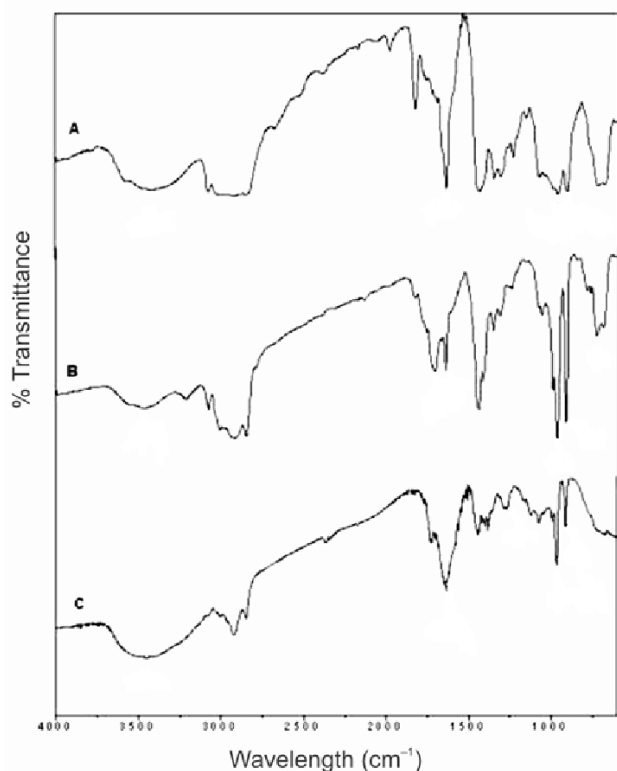


Figure 4. Comparative attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectra for (a) Cu, (b) Cu-Zn and (c) Cu-Zn-Sb (photo shows vibrational spectra for (a) Cu, (b) Cu-Zn and (c) Cu-Zn-Sb)

As ray emitted from samples is used to affect cancer cells, its amount was measured through selecting optimum concentration of semiconductor on the produced samples and concentration of required Tris(2,2'-bipyridyl)ruthenium(II) (Ru(bpy)₃2⁺) solvent before applying synchrotron on cancer cells. In this regard, the intensity of synchrotron of samples in solvents with various concentrations were measured to find optimum concentration of semiconductor in the produced samples for a constant concentration of solvent, as shown in Figure (5). From this test, optimum amount of Copper Zinc Antimony Sulfide, CZAS

(Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor semiconductor was determined as 2 (mg/ml) and then, samples with optimum concentration of semiconductor were tested at different concentrations of Tris(2,2'-bipyridyl)ruthenium(II) (Ru(bpy)₃2⁺) solvent and again, optimum synchrotron was obtained as 20 (mM). Figure (6) indicates this optimum amount.

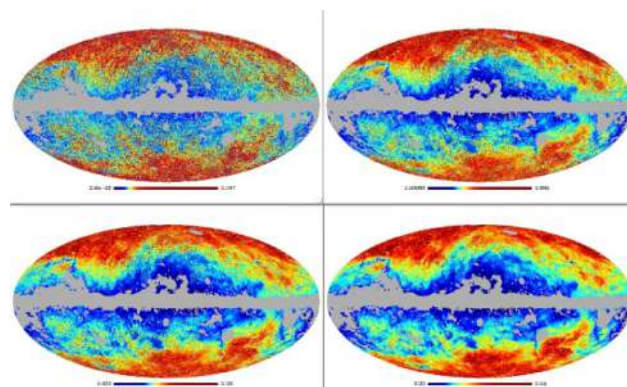


Figure 5. Photon emission for various concentrations of semiconductor (Figure: Optimization graphs of concentration of Copper Zinc Antimony Sulfide, CZAS (Cu_{1.18}Zn_{0.40}Sb_{1.90}S_{7.2}) semiconductor semiconductor)

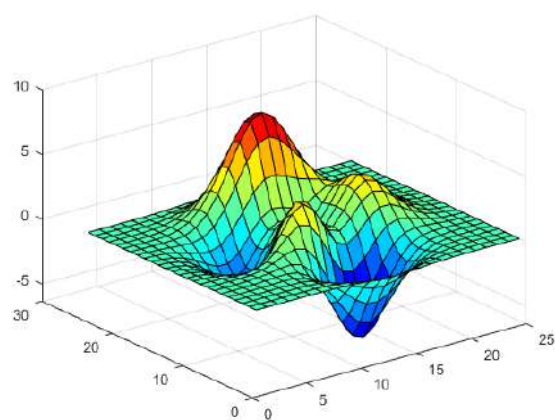


Figure 6. Photon emission for various concentrations of Tris(2,2'-bipyridyl)ruthenium(II) (Ru(bpy)₃2⁺) (Figure: Optimization graph of concentration of Tris(2,2'-bipyridyl)ruthenium(II) (Ru(bpy)₃2⁺))

4. Conclusions, Summary, Perspectives, Useful Suggestions and Future Studies

As the manufacture of synchrotronic biosensor is performed for the first time in the country, it was necessary to provide appropriate conditions such as high sensitivity and optimizing the effective factors in tracking, monitoring, imaging, measuring, diagnosing and detecting cancer

cells before any measurement. Lack of these conditions will lead to loss of cancer cells. Cancer is a major cause of death with more than 10 million annual patients. It is possible that this number reaches 15 million patients per year by 2020. Though chemotherapy has largely been successful in controlling and treating cancer, live tissue damage, systemic toxicity and side effects in this method are among the issues that cannot be overlooked. In order to reduce the negative effects of anticancer drugs on normal tissues, we need to design Nano-sized carriers that can pass the safety barriers and body tissues and reach their target site. In this research, the size and zeta potential of Nano-carriers PLGA-Cs-Paclitaxel were evaluated. Chitosan connection in physical or conjugated forms may lead to a significant increase of polydispersity. According to the study carried out on the concentration of Chitosan and the type of absorption, it was concluded that nanoparticles size increases with higher concentrations of Chitosan. The zeta potential will increase, provided the conjugation of Chitosan is higher than physical adsorption.

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This document provides some guidelines to authors for submission in order to work towards a seamless submission process. While complete adherence to the following guidelines is not enforced, authors should note that following through with the guidelines will be helpful in expediting the copyediting and proofreading processes, and allow for improved readability during the review process.

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Declaration

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Examples of conflicts of interest include (but are not limited to):

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A general introduction to the research topic of the paper should be provided, along with a brief summary of its main results and implications. Kindly ensure the abstract is self-contained and remains readable to a wider audience. The abstract should also be kept to a maximum of 200 words.

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Section headings, sub-headings, and sub-subheadings should be differentiated by font size.

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In this section, the results of experiments conducted should be detailed. The results should not be discussed at length in

this section. Alternatively, Results and Discussion can also be combined to a single section.

VIII. Discussion

In this section, the results of the experiments conducted can be discussed in detail. Authors should discuss the direct and indirect implications of their findings, and also discuss if the results obtain reflect the current state of research in the field. Applications for the research should be discussed in this section. Suggestions for future research can also be discussed in this section.

IX. Conclusion

This section offers closure for the paper. An effective conclusion will need to sum up the principal findings of the papers, and its implications for further research.

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XI. Glossary of Publication Type

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Conflicts of interest, acknowledgements, and publication ethics should also be declared in the final version of the manuscript. Instructions have been provided as its counterpart under Cover Letter.

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