KEMENTERIAN PENDIDIKAN MALAYSIA

FINAL REPORT
FUNDAMENTAL RESEARCH GRANT SCHEME (FRGS)

Laporan Akhir Skim Geran Penyelidikan Fundamental (FRGS)
Pindaan 1/2015

A **RESEARCH TITLE: SYNTHESIS AND CHARACTERIZATION OF ONE DIMENSIONAL MODIFIED COBALT NANOWIRES FOR NANOSCALE MECHANICAL**

PHASE & YEAR: PHASE 1 2016 (FRGS/1/2016/STG07/UMP/02/6)

START DATE: 1/08/2016
END DATE: 31/7/2018
EXTENSION PERIOD (DATE): RMC LEVEL: 31/1/2019
KPM LEVEL:

PROJECT LEADER: ASSOC. PROF. IR. TS. DR. MAHENDRAN SAMYKANO
I/C / PASSPORT NUMBER: 781123-04-5327

PROJECT MEMBERS: (including GRA)
1. PROF. DR. MD. MUSTAFIZUR RAHMAN
2. ASSOC. PROF. IR. TS. DR. KUMARAN KADIRGAMA
3. PROF. DR. JOSE RAJAN
4. ASSOC. PROF. TS. DR. DEVARAJAN RAMASAMY
5. TS. DR. MUHAMAD MAT NOOR
6. DR. NURUL AKMAL CHE LAH
7. DR. POORIA PASBAKHSH
8. JEEVENDRAN KANANATHAN (GRA)

PROJECT ACHIEVEMENT *(Prestasi Projek)*

B

ACHIEVEMENT PERCENTAGE			
Project progress according to milestones achieved up to this period	**0 - 50%**	**51 - 75%**	**76 - 100%**
Percentage (please state #%)			100%

RESEARCH OUTPUT		
Number of articles/ manuscripts/ books *(Please attach the First Page of Publication)*	**Indexed Journal**	***Non-Indexed Journal***
	3 ISI	
Conference Proceeding *(Please attach the First Page of Publication)*	**International**	***National***
	3 SCOPUS	
Intellectual Property *(Please specify)*	Nil	

P - 6 Index
Talent - Inc

UMP

HUMAN CAPITAL DEVELOPMENT

Human Capital	Number				Others (please specify)
	On-going		Graduated		
Citizen	Malaysian	Non Malaysian	Malaysian	Non Malaysian	
No. PHD STUDENT					
Student Fullname: IC / Passport No: Student ID:					
No. MASTER STUDENT			1		
Student Fullname: IC / Passport No: Student ID:			K. Jeevendran 900929106095 MMM16010		
No. UNDERGRADUATE STUDENT					
Student Fullname: IC / Passport No: Student ID:					
Total			1		

EXPENDITURE *(Perbelanjaan) as Borang K1(RMC)*

C

Budget Approved *(Peruntukan diluluskan)* : **RM 69,800.00**
Amount Spent *(Jumlah Perbelanjaan)* : **RM 67,367.97**

Balance *(Baki)* : **RM 1,432.03**
Percentage of Amount Spent : 99 %
(Peratusan Belanja)

ADDITIONAL RESEARCH ACTIVITIES THAT CONTRIBUTE TOWARDS DEVELOPING SOFT AND HARD SKILLS
(Aktiviti Penyelidikan Sampingan yang menyumbang kepada pembangunan kemahiran insaniah)

D

International

Activity	Date (Month, Year)	Organizer
ANM 2018 - 11TH INTERNATIONAL CONFERENCE ON ADVANCED NANO MATERIALS	18 July 2018 to 20 July 2018	University of Aveiro, Portugal

National

Activity	Date (Month, Year)	Organizer
Nil	Nil	Nil

E PROBLEMS / CONSTRAINTS IF ANY *(Masalah/ Kekangan sekiranya ada)*

UMP

Delay in obtaining the materials for the study and also delay in performing the characterization due to equipment mulfuction.

F **RECOMMENDATION** (*Cadangan Penambahbaikan*)

Findings shows a good possibility to futher this study for depth understandings on the behavior of the cobalt nanowire.

G **RESEARCH ABSTRACT – Not More Than 200 Words** (*Abstrak Penyelidikan – Tidak Melebihi 200 patah perkataan*)

The present research and dissertation focuses on: 1. Experimental synthesis of electrodeposited Co nanowires at different current densities and external magnetic fields, 2. Physical properties characterization of the synthesized nanowires to understand their morphology, structural and crystallographic properties. Key research insights from the present experimental research include: Electrodeposition method consistently synthesizes high purity Co nanowires (98% and higher based on energy dispersive spectroscopy (EDS)) with a significant improvement in surface morphology when magnetic field is present during synthesis; X-ray diffraction (XRD) characterization and analysis indicate that electric current density has significant influence on the crystal orientation of Co nanowire, while a decrease in crystal size was noticed with increased magnetic field intensity for same current densities.

Date : 1/10/2019
Tarikh

Project Leader's Signature:
Tandatangan Ketua Projek

H **COMMENTS, IF ANY/ ENDORSEMENT BY RESEARCH MANAGEMENT CENTER (RMC)**
(Komen, sekiranya ada/ Pengesahan oleh Pusat Pengurusan Penyelidikan)

The overall performance is excellent.
Achieved KPI
Recommended to close this project

Name:
Nama:

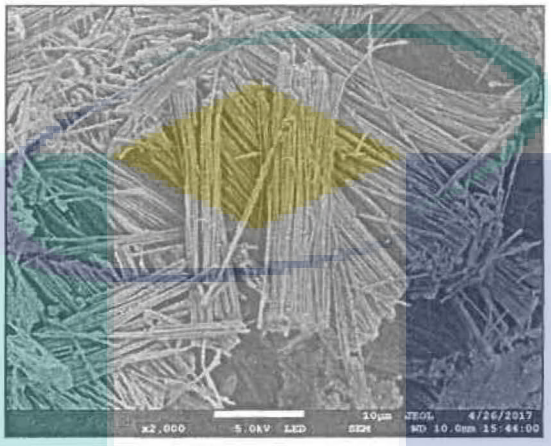
Signature:
Tandatangan:

Date:
Tarikh: 14/10/19

PROFESOR Ts. DR. MD MUSTAFIZUR RAHMAN
Dekan Penyelidikan
Jabatan Penyelidikan dan Inovasi
Universiti Malaysia Pahang

UMP

TEMPLATE
BUKU PROFIL PENYELIDIKAN SKIM GERAN PENYELIDIKAN FUNDAMENTAL (FRGS) FASA 1/2016



SYNTHESIS AND CHARACTERIZATION OF ONE DIMENSIONAL MODIFIED COBALT NANOWIRES FOR NANOSCALE MECHANICAL DEVICES

Name of Project Leader
ASSOC. PROF. IR. TS. DR. MAHENDRAN SAMYKANO

Name of co-researchers
PROF. DR. MD. MUSTAFIZUR RAHMAN
ASSOC. PROF. IR. TS. DR. KUMARAN KADIRGAMA
PROF. DR. JOSE RAJAN
ASSOC. PROF. TS. DR. DEVARAJAN RAMASAMY
TS. DR. MUHAMAD MAT NOOR
DR. NURUL AKMAL CHE LAH
DR. POORIA PASBAKHSH

IPT/ Faculty / School/ Centre/Unit
E-mail
FRGS Field

ABSTRACT (120 words)

The present research and dissertation focuses on: 1. Experimental synthesis of electrodeposited Co nanowires at different current densities and external magnetic fields, 2. Physical properties characterization of the synthesized nanowires to understand their morphology, structural and crystallographic properties. Key research insights from the present experimental research include: Electrodeposition method consistently synthesizes high purity Co nanowires (98% and higher based on energy dispersive spectroscopy (EDS)) with a significant improvement in surface morphology when magnetic field is present during synthesis; X-ray diffraction (XRD) characterization and analysis indicate that electric current density has significant influence on the crystal orientation of Co nanowire, while a decrease in crystal size was noticed with increased magnetic field intensity for same current densities.

1. INTRODUCTION

Recent experimental observations have shown that the properties of nanowires are strongly influenced by processing methods and synthesis parameters employed. Of the various methods cited in literature (Xia et al., 2003); electrodeposition is shown to be a viable and effective method for Ni nanowires (Samykano, Mohan, & Aravamudhan, 2015). In template-assisted electrodeposition method, process parameters such as solution pH, temperature, agitation condition, template material, additive, and current density are found to determine the properties of the nanowires synthesized. Additionally, as Co is one of the four ferromagnetic elements which have ferromagnetic properties - application of magnetic field is found to influence its properties. Literature cites extensive work on the influence of magnetic field on the magnetic properties of electrodeposited Co nanowires. However, limited work to date has focused on understanding physical and mechanical property changes associated with application of external magnetic field, and variation in current density during electrodeposition. Present research focuses on such understanding; striving to develop a potential correlation between processing, physical, and mechanical properties through careful experimental design, experimentation, and analysis.

However, due to the complications and challenges associated with mechanical testing at nanoscale, very little attention has been given to experimentally characterize polycrystalline nanowires, and mechanical properties of the polycrystalline nanowires are still not very well understood (Tian et al., 2010). From a nanotechnology applications perspective, such understanding of structural-mechanical properties are very critical to avoid mechanical failure due to incidental forces in nano-devices (Varghese et al., 2008).

The specific research objectives

- Electrodeposition synthesis of Co nanowires
- Physical property characterization and analysis.
- Analyze experimental data obtained to study and understand the influence of electric current density and external magnetic field on physical properties of synthesized Co nanowires.

2. RESEARCH METHODOLOGY

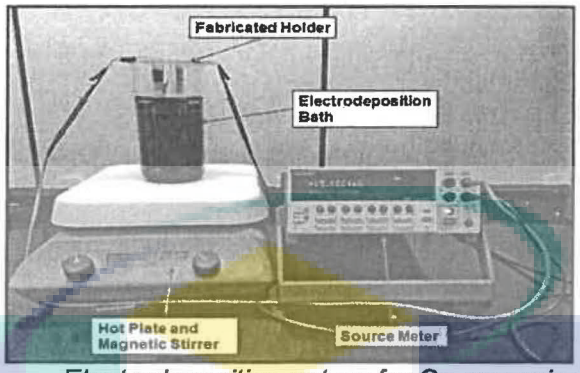


Figure 1 Electrodeposition set-up for Co nanowire synthesis

The nanowire synthesis is performed either with or without magnetic field. For nanowire synthesis with magnetic field, known magnetic field intensity was placed close to the template and electrode (with magnetic field aligned parallel to nanowire growth). As the stoichiometry of the Co nanowires are significantly affected by solution pH, agitation conditions, temperature, external magnetic field, solution additives and deposition time, all these process parameters were maintained to be consistent during the electrodeposition process except for the external magnetic field and current density.

Table 1 lists the deposition parameters used in the present work. All synthesis was done for 1 hour of deposition at a temperature of about 35 °C. After the electrodeposition process, the templates were cleaned with deionized water prior to dissolution in sodium hydroxide (NaOH) solution to obtain freestanding Co nanowires. NaOH solution was later replaced with methanol solution.

Table 1 Deposition process parameters used for synthesis

Parameters	Values	Units
Magnetic field	0, 3817 and 5758	Gauss
Current density	3, 5 and 7	$mA.cm^{-2}$
Temperature	35	°C
Solution pH	4.2 - 4.8	n/a
Agitation	100	rpm
Time	60	minutes

Physical properties characterization of individual nanowires is a challenge to many current testing and measuring techniques. These are due to their size which is tiny, prohibiting the applications of the well-established characterization techniques. SEM (Carl Zeiss, Auriga) is used to study the structure and morphology of the grown Co nanowires. This study was performed by qualitative observation of the surface of the electrodeposited Co nanowires from a series of images taken using SEM. The EDS analysis is performed using Oxford Instruments EDS attached to Carl Zeiss EVO SEM at a beam source of 15 kV. A quantitative EDS analysis is performed on all the samples to determine the elemental composition of the Co nanowires deposited in the AAO template at different synthesis conditions. X-ray diffraction experiments were performed on Gemini XRD (Keysight Technologies) with a monochromatized Cu Kα (λ = 0.154 nm) radiation source operated at 40 kV and 40 mA in a powder diffraction arrangement.

3. LITERATURE REVIEW

One-dimensional (1-D) nanostructures are slivers of material that are constrained in other 2 directions. Within this category of nanomaterials, researchers differentiate between nanorods that are generally with aspect ratios less than 10, nanowires with aspect ratios greater than 10 and nanotubes with hollow interior (Chen, Wiley, & Xia, 2007). In the past two decades, 1-D nanostructures have got serious attention, with several researchers studying numerous methods to synthesize 1-D nanostructures with different shapes, morphology, size and materials with exciting and fundamentally different configurations (Barth, Hernandez-Ramirez, Holmes, & Romano-Rodriguez, 2010; Kuchibhatla, Karakoti, Bera, & Seal, 2007; C. Rao, Deepak, Gundiah, & Govindaraj, 2003; N. Wang, Cai, & Zhang, 2008).

Among above mentioned strategies, growth dictated by the use of a template has been widely employed. Various template-assisted methodologies had been shown to be successful to synthesize 1-D nanostructures. Among them are: step edges present on the surfaces of a solid substrate (Walter et al., 2002), channels in porous material (Huczko, 2000), self-assembled molecular structures (Murphy & Jana, 2002) and templating against existing nanostructures (Sun, 2010). Although these prior techniques have shown to be successful, the technique that employs channels in porous material seems to more preferred and extensively used.

Technique for synthesis of 1-D nanostructures using porous material was initiated by Martin and several others (Hulteen & Martin, 1997; Martin, 1996). The porous material technique has been used in conjunction with various processing methods such as electrochemical deposition (Brumlik, Menon, & Martin, 1994), sol-gel (Z. Wang et al., 2011), chemical vapor deposition, CVD (Li et al., 2011) and pressure injection (Phillips, Fang, Zheng, & Lagoudas, 2011) to produce various types of 1-D nanostructures with interesting properties.

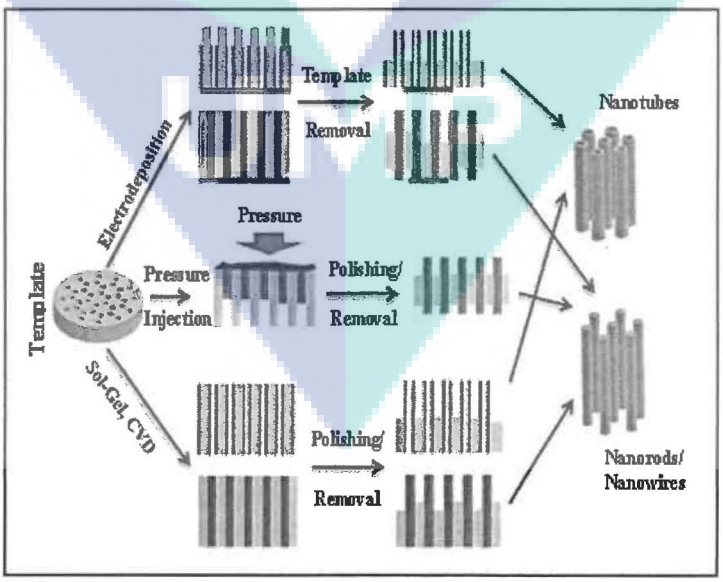


Figure 2 Flow diagram for template-assisted synthesis

4. FINDINGS

Scanning Electron Microscope Analysis

SEM analsys show that the surface texture and roughness is found to be drastically reduced as the magnetic field strength is increased. The surface texture changed from very rough when no magnetic field is applied to a smooth surface at the highest magnetic field. This is due to magnetohydrodynamic (MHD) effect from the applied magnetic field which increases the limiting internal potential and deposition rate. Thus the deposits obtained with magnetic field appear to be smoother and uniform compared to that without magnetic field.

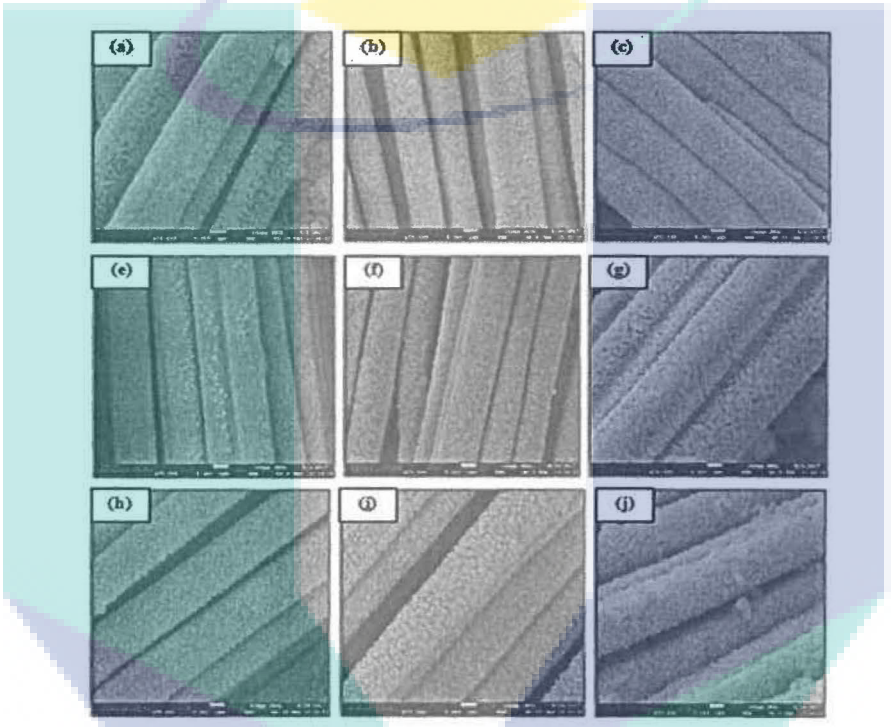


Figure 3 *SEM images of Co nanowires synthesized at different current densities with the absence and presence of magnetic field*

The growth length of Co nanowires is found to increase significantly as the current density is increased. The resultant significant increase in the growth can be attributed to higher overpotential due to increase in the applied current density which subsequently increases the nucleation rate (Rashidi & Amadeh, 2008; Schmickler & Santos, 2010) and ionic mass transfer rate within the nanosized diameter pore (Konishi et al., 2003; Motoyama, Fukunaka, Sakka, & Ogata, 2006).

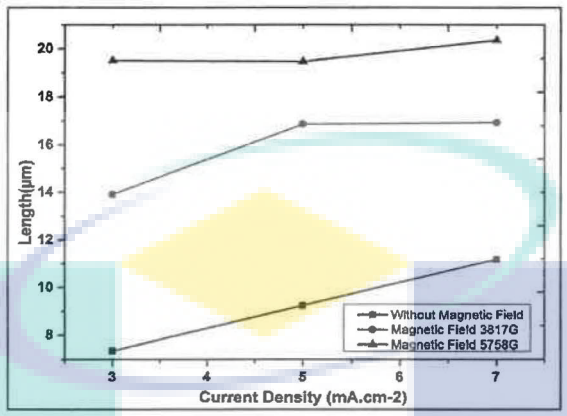


Figure 4 Co nanowire grown length at different proses condition

Energy Dispersive Xray Spectroscopy

The EDS analysis demonstrates that the synthesized nanowires were consistently composed of 96.73±0.66 % of Co and 3.27±0.66 % of oxygen. The small amount of oxygen observed in the entire spectrum indicates potential absorption from air on the surface of the nanowires.

Table 2 EDX elemental composition of synthesized Co nanowires

Elements	Value (%)	Standard deviation (%)
Cobalt (Co)	96.73	0.66
Oxygen (O)	3.27	0.66

X-ray powder diffraction Analysis

Present XRD results show there was not a significant influence of magnetic field on the orientation of the crystals. The predominant orientation is still dictated by the electric field. The preferred orientation of synthesized Co nanowires in all cases was found to be (1 0 0) plane, followed by (0 0 2) and (1 0 1) planes.

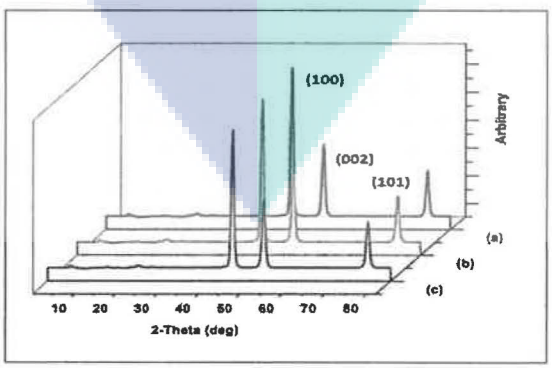


Figure 5 XRD spectrum of Co nanowires synthesized at different current density with the absence and presence of magnetic field

Crystal size analysis indicate that the average crystal size reduces upon application of magnetic field with all three different current densities. It is also noted that the crystal size decreases and then increases as the current density is increased in the absence of magnetic field. This needs further investigation to understand the rationale for the formation of inconsistent crystal size as the current density increased. Even though there is inconsistent crystals size formation as the current density is increased, upon application of magnetic field, the crystal size is found to be decreased but with no significant differences. The resultant decrease of this crystal size can be attributed to the coupled effect of current density and magnetic field known as magnetohydronamic effect (MHD)

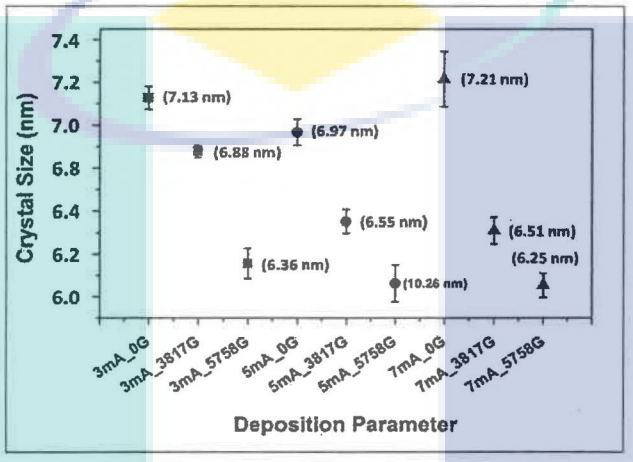


Figure 6 Crystal size of Co nanowires synthesized at different current density with the absence and presence of magnetic field

5. CONCLUSION

Co nanowire was successfully synthesized using template-assisted electrodeposition technique consistently and with good repeatability. The diameter of the nanowire was found to be between 200 to 36 nm in diameter based on the variations in the pore size of the template used. EDS analysis showed the synthesized structure was composed of 96.73% of Co and 3.27% of oxygen. The observed oxygen is primarily due to absorption from air on the surface of the nanowires after synthesis.Qualitative SEM image analysis has shown that the surface roughness improved as the intensity of the magnetic field increased, while morphology changes due to increased current density are found to be not very significant. Overall growth length is also found to be significantly improved as the current density increased since the deposition rate is primarily dictated by current density. Application of magnetic field further improves the deposition rate as an improved growth length obtained.The existence of multiple planes from XRD spectra suggested that the synthesized Co nanowires were polycrystalline in nature. The crystal orientation of synthesized Co nanowires at all investigated processing conditions were found to be preferred at (1 0 0) plane followed by (0 0 2) and (1 0 1) plane. The orientation of the crystal was found to be influenced by magnetic field. The predominant orientation was found to be dictated primarily by current density. The measurement of crystal size from XRD experiments shows that the average crystal size decreases as the intensity of magnetic field increased. Inconsistent formation of crystal size was observed when the current density increased.

ACHIEVEMENT

i) Name of articles/ manuscripts/ books published

1. Mechanical properties–Theory and experiment. Applied Materials Today, 11, 320-337.
2. An overview of marine macroalgae as bioresource. Renewable and Sustainable Energy Reviews, 91, 165-179.
3. Influence of boric acid (H3BO3) concentration on the physical properties of electrochemical deposited nickel (Ni) nanowires. In IOP Conference Series: Materials Science and Engineering (Vol. 257, No. 1, p. 012033). IOP Publishing
4. Electrochemical deposited nickel nanowires: influence of deposition bath temperature on the morphology and physical properties. In IOP Conference Series: Materials Science and Engineering (Vol. 257, No. 1, p. 012032). IOP Publishing.
5. Plasmonic behaviour of phenylenediamine functionalised silver nanoparticles. Materials Research Express, 4(9), 095018.
6. Nanofluid as coolant for grinding process: An overview. In IOP Conference Series: Materials Science and Engineering (Vol. 342, No. 1, p. 012078). IOP Publishing.

ii) Title of Paper presentations (international/ local)
Nickel and Cobalt Nanowire: Magnetic Ordering Synthesis at International Conference On Advanced Nano Materials (ANM, 2018), University of Aveiro, Portugal

iii) Human Capital Development
One (1) Master Student (Graduated), Jeevendran Kananathan, MMM16010.

iv) Awards/Others
Nil

v) Others
Nil

REFERENCES

[1] Alkire, R. C., Gogotsi, Y., Simon, P., & Eftekhari, A. (2008). *Nanostructured Materials in Electrochemistry*. Weinheim: Wiley-VCH.
Bard, A. J., & Faulkner, L. R. (1980). *Electrochemical methods: fundamentals and applications* (Vol. 2): Wiley New York.

[2] Barth, S., Hernandez-Ramirez, F., Holmes, J. D., & Romano-Rodriguez, A. (2010). Synthesis and applications of one-dimensional semiconductors. *Progress in Materials Science, 55*(6), 563-627.

[3] Brumlik, C. J., Menon, V. P., & Martin, C. R. (1994). Template synthesis of metal microtubule ensembles utilizing chemical, electrochemical, and vacuum deposition techniques. *Journal of Materials Research, 9*(5), 1174-1183.
Cao, G. (2004). *Nanostructures & Nanomaterials: Synthesis, Properties & Application*. London: Imperial College Press.

[4] Cao, G. Z., & Liu, D. W. (2008). Template-based synthesis of nanorod, nanowire, and nanotube arrays. *Advances in Colloid and Interface Science, 136*(1-2), 45-64. doi:DOI 10.1016/j.cis.2007.07.003

[5] Chen, J., Wiley, B. J., & Xia, Y. (2007). One-dimensional nanostructures of metals: large-scale synthesis and some potential applications. *Langmuir, 23*(8), 4120-4129.

[6] Coey, J., & Hinds, G. (2001). Magnetic electrodeposition. *Journal of alloys and compounds, 326*(1), 238-245.

[7] Cortes, A., Riveros, G., Palma, J. L., Denardin, J. C., Marotti, R. E., Dalchiele, E. A., & Gomez, H. (2009). Single-crystal growth of cobalt nanowires: influence of deposition conditions on structural and magnetic properties. *Journal of Nanoscience and Nanotechnology, 9*(3), 1992-2000. doi:Doi 10.1166/Jnn.2009.374

[8] Djokić, S. S., & Cavallotti, P. L. (2010). Electroless Deposition: Theory and Applications *Electrodeposition*. New York: Springer.

[9] Huczko, A. (2000). Template-based synthesis of nanomaterials. *Applied Physics A- Materials Science & Processing, 70*(4), 365-376. doi:DOI 10.1007/s003390051050

[10] Hulteen, J. C., & Martin, C. R. (1997). A general template-based method for the preparation of nanomaterials. *Journal of Materials Chemistry, 7*(7), 1075-1087. doi:Doi 10.1039/A700027h

[110 Kamalakar, M. V. (2011). *Synthesis, Characterization and Investigation of Electrical Transport in Metal Nanowires and Nanotubes*. (PhD), Kolkata, India.

[12] Kelsall, R. W., Hamley, I. W., Geoghegan, M., & Wiley, J. (2005). *Nanoscale Science and Technology*. Weinheim: Wiley-VCH.

UMP

SENARAI DAFTAR HARTA MODAL DI BAWAH DANA PENYELIDIKAN FUNDAMENTAL KEMENTERIAN PENDIDIKAN TINGGI

BIL	IPT	JENIS GERAN (FRGS/ERGS/LRGS/PRGS/NRGS/RAGS/RACE/TRGS)	FASA (CONTOH 2/2014)	TAJUK PROJEK PENYELIDIKAN	NAMA KETUA	NAMA ASET	BILANGAN UNIT	NO SIRI PENDAFTARAN	TARIKH PEROLEHAN	HARGA PEROLEHAN ASAL (RM)
1	UMP	FRGS (RDU160119)	Fasa 1 / 2016	SYNTHESIS AND CHARACTERIZATION OF ONE DIMENSIONAL MODIFIED COBALT NANOWIRES FOR NANOSCALE MECHANICAL	Assoc. Prof. Ir. Ts. Dr. Mahendran Samykano	Tiada	Tiada	Tiada	Tiada	Tiada
2										
3										
4										
5										

UMP