A

DISSERTATION REPORT

ON

"Friction stir welding of aluminium alloys"

Submitted by

Karale Tejal Ramchandra

(MIS No: 121226018)

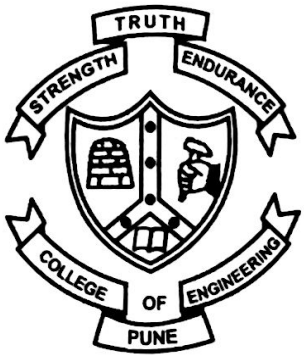
In partial fulfillment for the award of degree of

M.Tech (Physical Metallurgy)

In

METALLURGY AND MATERIALS SCIENCE

Under The Guidance of
Prof. M. J. Rathod



DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING PUNE – 411005

(An autonomous institute of Government of Maharashtra)

2013-2014

Abstract

Friction stir welding (FSW) is a solid state welding process specially used for joining nonferrous alloys like aluminium alloys, magnesium alloy, copper alloys and titanium alloys. FSW has various advantages over conventional welding method like low distortion of material, lower loss of alloying materials, high strength of joint, energy efficient, environment friendly, and versatile. Present study was aimed to understand microstructural changes and associated mechanical properties of the FSW joints of similar metals sheets of aluminium alloys - Al6061 and Al5052. The same alloy pair was welded by FS W to form dissimilar joints. Mainly the effect of tool geometry namely - conical pin and hexagonal pin – was studied. Operating parameters - tool rotational speed and traverse speed were changed by keeping tilt angle and plunge depth constant. The welded joints were tested for tensile properties and impact toughness. Metallographic observations of joints were carried out using optical microscopy. Hardness profiles were determined and residual stress was measured in transverse direction. The optimum process parameters were determined for tensile strength of the joints.

For joining of Al6061:Al6061 the optimum process parameters were 1400 rpm rotational speed and 100 mm/min travel speed with hexagonal pin tool. With rotational speed of 1150 rpm, travel speed of 60 mm/min and conical tool optimum properties of Al5052:Al5052 were achieved. For dissimilar materials joints of Al6061:Al5052 made with hexagonal tool at tool speed 900 rpm and travel speed 100 mm/min resulted in optimum tensile strength. The nugget zone mostly showed fine equiaxed grains. Hardness values at the stirred zone were lower than those in the parent material for all three joints. Impact strength values of the FSW joints were found to be lower than those of parent materials. Maximum impact energy was observed with optimum process parameters. Compressive residual stresses were found to be induced in the weld nugget and tensile residual stresses were noticed in the HAZ.

Dissertation Report
on

"Analysis of Thermal Fatigue Behavior of H13 Hot Work Tool Steel"

Submitted By

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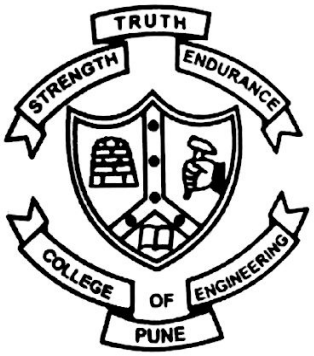
In partial fulfilment for the award of degree

of

M.Tech (Physical Metallurgy)

METALLURGY AND MATERIALS SCIENCE

Under The Guidance of
Prof S.U.DANGRIKAR



DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING, PUNE – 411005

(An autonomous institute of Government of Maharashtra)
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ABSTRACT

Casting industries have a very huge contribution among manufacturing sector. Die casting is a choice for low melting point alloys like Aluminium, Zinc, and Magnesium. There are about 33 processes commercially available for the production of aluminium castings and about 70% of the castings are produced using conventional high-pressure die-casting process. Die casting is a high volume production process hence downtime, breakdown due to die related problems results in loss of production rate and cost. Dies cost about 20% of the total cost of producing aluminium die castings.

Dies for aluminium alloys die-casting fail because of a great number of a different and simultaneously operating factors. Die design, material selection, and thermal stress fatigue due to the cyclic working process, as well as to low and inhomogeneous initial die temperature contribute to the failures and cracks formation on/in dies. Thermal fatigue cracking is a major mode of failure of hot working tool steel dies in die-casting. The cracking initiates due to the large thermal shock experienced by the die surface when it is rapidly heated to 700 °C and then quenched to 150 °C by the lubricant spray. This thermal fatigue loading propagates the crack until gross failure occurs or die becomes unusable.

X-ray diffraction (XRD) stress measurement can be a powerful tool for failure analysis or process development studies. Quantifying the residual stresses present in a component, which may either accelerate or arrest thermal fatigue, is frequently crucial to understanding the cause of failure. Die material for aluminium die-casting needs to be resistant to heat checking, gross cracking, soldering and washout. To resist heat checking, die materials should have a higher strength and toughness, low coefficient of thermal expansion, high thermal conductivity, and resistance to temper softening.

In practice, H13 steel is the most popular material for aluminium die casting dies. Immersion test apparatus was developed, which enables simulation of conditions during aluminium alloy die casting and enables controlled thermal fatigue testing of materials. Special specimens made of the AISI H13 tool steel were used for thermal fatigue test. Heat treatment on the mechanical properties of H13 hot working die steel for die casting is discussed.

The heat treating industry is currently processing dies in vacuum furnaces utilizing nitrogen gas quenching. A faster cooling rate can be attained with 6-7 bar gas quenching and this can be accomplished without objectionable distortion. The steel was treated by austenitising at 1030°C and quenched at cooling rates of 30, 43 and 56°C/min followed by

multiple tempering to investigate its mechanical properties. Induced microstructures are revealed using optical microscope and phase compositions are assessed by means of X-ray diffraction technique while mechanical characteristics are investigated based on hardness and toughness standard tests.

After heat treatment, material exhibits the tempered martensitic structure with wider laths of martensite connected to a complex carbides mainly of M23C6 and M7C3 chromium carbides type. The intense martensite tempering is observed on the die surface, while the core material remains unchanged. A considerable hardness change is measured from the material surface to core up to three millimetres distance. Toughness property is found to be higher for increasing quenching rate from slower to higher. Results of residual stress analysis shows the dependency of tool manufacturing process and thermal fatigue conditions. Lower thermal gradients keeps the stresses in lower level.

DISSERTATION

ON

THERMAL AND TRIOBOLOGICAL PROPERTIES OF POLYARYLETHERKETONE (PAEK) BASED MICRO- AND NANOCOMPOSITES

Submitted by

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For

Partial Fulfillment of

M.Tech (PHYSICAL METALLURGY)

Under the guidance of

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DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING, PUNE

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ABSTRACT

Polymer matrix micro- and nanocomposites are widely used in tribological applications due to their easy processing and low wear rate. In this work, polymer matrix composite based on poly(aryletherketone) (PAEK) as matrix and micro molybdenum disulphide (MoS_2) and nano boron nitride (BN) powders as reinforcement were fabricated using planetary ball milling process under dry condition followed by hot pressing. The MoS_2 and BN content was varied from 0.5 wt % to 5 wt%. Both powders were mixed at room temperature for 5 h at 250 rpm. Experimental density of the composites was measured using Archimedes principle. The experimental density of the composites was close to the theoretical density indicating porosity free samples. The X-ray diffraction showed that PAEK is semicrystalline polymer. Thermogravimetric analysis showed enhanced thermal stability and char yield on increasing the MoS_2 and BN particles in the PAEK matrix. The microhardness of the PAEK micro- and nanocomposites increases with increasing MoS_2 and BN content. The wear rate of the composites was determined using novel variable loading wear and friction monitor (VLWAFM) pin-on-disk tribometer in dry condition. The wear rate of both the composites were evaluated at a sliding speed of 1.0 m/s and nominal pressure of 0.5 MPa to 3 MPa in static loading. The wear rate was reduced maximum by 31 times at 1 MPa load for 0.5 wt. % of PAEK/MoS_2 composite and 57 times at 0.5 MPa load for 0.5 wt. % of PAEK/BN nanocomposite. For dynamic loading wear rate reduced by half for 1 wt. % of PAEK/MoS_2 composite at varying load 0.5 MPa-1 MPa under dry sliding conditions. Based on above results, both composites can be used for tribological applications.

Study of Powder Forged Properties of Gear Material

A Project Report

Submitted by

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In partial fulfillment for the award of the degree

Of

M. TECH (PHYSICAL METALLURGY)

IN

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING, PUNE

(An autonomous institute of Government of Maharashtra)

Under the guidance of

Prof. VIJAY THAVALE (guide)

Dr. N.B. DHOKEY (co-guide)

July 2014

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

Abstract

Gears for engineering applications are normally manufactured by forging a blank, turnings, hobbing, shaving and heat treatment followed by grinding. Due to the size of the gear the net shape capability of powder metallurgy methods can be cost effective in place of conventional manufacturing. In the proposed research, the standardization of compaction and sintering parameters viz. sintering temperature, holding time and compaction pressure were carried out. Sintering at higher temperature (1120 °C) was employed for Astaloy Mo in reducing atmosphere tubular furnace using a gas mixture of N_2/H_2 (90/10 by % volume). Powder forging process was adopted to achieve the higher densification. The density distribution was studied, by forging as a function of % height reduction with open die and close die forging. Mechanical properties were characterized such as density, hardness and microstructure. Compaction pressure of 600 MPa and forging at 600 °C with 50% height reduction was optimized which gave highest density of 98% of the theoretical density and hardness of 95 HRB. As optimum Sintering-Forging-Heat treatment cycle was established.

Key words: Forging, Hardness, Density, Gear materials, Sintering, Compaction

A

DISSERTATION REPORT

ON

"Synthesis of $ZnNb_2O_6$ Nano-ceramics, Polycarbonate-$ZnNb_2O_6$ Composites and their Dielectric Properties"

Submitted by

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In partial fulfillment for the award of the degree

of

MASTER OF TECHNOLOGY

in

Physical Metallurgy

Under the Guidance of

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Year-2012-13

Abstract

Nano-powders of $Zn_xNb_2O_6$ (x=0.95-1.05) and pure phase $ZnNb_2O_6$ ceramics were prepared by Co-precipitation (CPT) and Pechini technique respectively. The precipitation sequence of pure phase $ZnNb_2O_6$ during co-precipitation was studied by characterizing the powders retained on two different filter papers of sizes 41 and 44 number and those which have remained suspended in the solution after filtration. The samples were analyzed for XRD (done after pre-firing the powders at 700° C for 2h), SEM and TG-DTA. The SEM microphotographs of Co-precipitated powders demonstrated almost circular particle morphology and an average powder particle size of 50nm. XRD results of samples pre-fired at 700°C showed crystalline $ZnNb_2O_6$ phase formation associated with peaks of either Nb_2O_5 or ZnO. TGA results demonstrated higher weight loss and DTA results exhibited peaks of desolvation reactions for the filtered particles (analyzed after drying). Interestingly, instead of an expected co-precipitation of ZnO plus Nb_2O_5 powders, it was observed that the reaction rather involved rapid initial precipitation of Nb_2O_5 particles characterized by its more initial occurrence, followed by relatively finer ZnO powder precipitation occurring sluggishly towards the end.

The powders were subsequently subjected to calcination and sintering to obtain dense single columbite phase ceramics and their dielectric properties were studied. Minimum calcination temperature to form pure $ZnNb_2O_6$ in case of Pechini was 800°C, whereas, in case of CPT all the constituents did not react even after calcination at 900°C. By varying the compaction pressures and sintering temperatures, a density of almost 80 % maximum could be achieved in the ceramics pellets made using powders from both routes. Sintering temperature required for a sample made by Pechini was 1000°C, whereas, it was 1200°C for samples made by CPT route. The XRD pattern of CPT powders demonstrated single phase $ZnNb_2O_6$ only after sintering at 1200°C. Chemical compound was analyzed by XRD and dielectric properties were studied by precision impedance analyzer, which showed: $\varepsilon_r = 25.5$ and $\tan\delta = 0.02$ for $ZnNb_2O_6$ made by CPT technique and $\varepsilon_r = 16.3$ and $\tan\delta = 0.007$ for $ZnNb_2O_6$ made by Pechini technique. The highest dielectric constant value of 28.5 with $\tan\delta$ of 0.03 was obtained for $Zn_{1.05}Nb_2O_6$ phase.

The pure $ZnNb_2O_6$ calcinated powders obtained from both routes were also used to fabricate the polymer matrix composites by using them as fillers in polycarbonate (PC) matrix. $ZnNb_2O_6$ (ZN) was varied from 0 wt. % to 60 wt. % in the PC matrix. The dielectric and

microhardness properties of the composites made were studied. The dielectric constant of the composites measured at 1 MHz increased approximately to 18 and 10 in case of powders made by CPT and Pechini respectively as against a dielectric constant of 3 (at 1MHz) obtained for pure Polycarbonate. The dissipation factor of these composites varied in the range of 0.012 to 0.029. SEM microphotographs showed a uniform dispersion of ZN in the PC matrix up to 40 wt. % addition. The XRD of composites made using CPT powders showed multi-component phases, whereas, those made using Pechini powders showed pure ZN phase. The microhardness of the composites increased up to 24.2 Hv (for Pechini) and 21.6 Hv (for CPT) as compared to pure PC, which was 16 Hv. These results demonstrated the suitability of these composites as well as the ceramics pellets made as class one type dielectric materials for resonant circuit applications.

Keywords: Co-Precipitation, Dielectric Properties, Pechini, Polymer Matrix Composite and Zinc niobate ($ZnNb_2O_6$).

Dissertation report

On

""Effect of Cryotreatment on Aluminium 6061"

Submitted by

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In partial fulfillment for the award of the degree

Of

M. TECH (PHYSICAL METALLURGY)

In

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

Under the guidance of

Prof. Mrs. S. U. Dangrikar.

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING, PUNE-411005

(An Autonomous Institute of Government of Maharashtra)

July 2014

ABSTRACT

The ageing phenomenon in age hardenable Aluminium alloy is complex and well under research. The supersaturated solid solution of these alloys, during ageing, undergoes a complex precipitation causing increase in strength with time. These alloys have a wide range of application in the aviation and automotive industry. Cryogenic has been tried on tool and alloy steel where it has been found that this treatment positively affects the wear resistance and also causes uniformity in structure. The cryogenic treatment has become an important step in the manufacturing tool steel.

This project comprises of mixing these two processes i.e. Ageing and Cryotreatment to find out if the material shows any significant change in properties.

So here I have first solutionised the sample, quenched it, then done the cryotreatment for 24 hours. The samples were taken out at 4 hours each i.e. 4 Hrs, 8 Hrs, 12 Hrs, 16 Hrs, 20 Hrs and 24 Hrs. After the cryotreatment the samples are aged artificially at 185 °C (as Per ASTM), 150 °C and 200 °C to optimize the ageing temperature and then the characterization is done on the basis of Hardness, Tensile Test, XRD, Microstructures and Scanning Electron Microscopy (SEM). It was found that 12 hours of cryotreatment and the artificial ageing is the optimum for better properties.

A

DISSERTATION

ON

"SYNTHESIS AND CHARACTERISATION OF $CaTiO_3$ BASED DIELECTRIC CERAMICS"

Submitted by

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In partial fulfillment for the award of the degree
Of
MASTER OF TECHNOLOGY
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Physical Metallurgy

Under the Guidance of
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Year-2013-14

Abstract

Development of new dielectric ceramics for various applications like microwave, communication, transducers etc. is the need of the time. The present work involves the fabrication and characterization of substituted $CaTiO_3$ ceramic. The addition of various substitutions like Manganese (in place of Ca^{2+}), Zirconium and Tin (in place of Ti^{4+}) is expected to promote grain growth within the microstructure, resulting in a denser ceramic. Further, a distorted orthorhombic perovskite crystal structure is also expected due to substitutions. $Ca_xMn_{1-x}TiO_3$, $CaTi_xZr_{1-x}O_3$ and $CaTi_xSn_{1-x}O_3$, (x=0-0.20) ceramics were prepared by a conventional solid state reaction method. The powders thus obtained were compacted uniaxially at 100MPa and sintered at 1200°C for Mn substitution and 1300°C for Zr and Sn substitution for 3-4 hours. XRD revealed mostly single phase structure for all the substitutions made with traces of ZrO_2 phase. Presence of ZrO_2 particles was also noticed in SEM image of $CaTi_xZr_{1-x}O_3$ samples. The maximum achievable dielectric constant and quality factor for $Ca_xMn_{1-x}TiO_3$ samples were 79 and 2377, for $CaTi_xZr_{1-x}O_3$ samples those were 74 and 1915 and for $CaTi_xSn_{1-x}O_3$ samples a value of 83 and 5536 respectively was notes. A marginal variation in DC conductivity was noted in the entire range of substitutions made.

Dissertation Report

On

"Study of Surface Mechanical Attrition Treatment on Ferrous and Non ferrous alloys"

Submitted by

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In partial fulfillment for the award of the degree

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M. TECH (PHYSICAL METALLURGY)

IN

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

Under the guidance of

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DEPARTMENT OF METALLURGY AND MATERIAL SCIENCE

COLLEGE OF ENGINEERING, PUNE

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ABSTARCT

This project mainly aimed at studying the effects of Surface Mechanical Attrition Treatment (SMAT) on 6061-T6 Aluminium alloy, Duplex stainless steel (2205), & AISI 304L steel.

Generally SMAT is a surface modification technique in which there is repeated impact of balls (bearing steel) on surface due to which severe plastic deformation takes place leading to formation of nanocrystalline layer in top layer of surface (up to 50 μm from edge of surface). So grain refinement occurs, forming nano grains resulting in increase in hardness of surface.

In this project detailed study on the influence of SMAT on 6061-T6 Aluminium alloy, Duplex stainless steel (2205), & AISI 304L steel. SMAT changes various properties of material such as increase in hardness of material at the surface, change in phase occurs (transformation of austenite to martensite), increase in thermal stability of material, increase in corrosion resistance, wear resistance of material. Through different set of experiments many interesting results have been obtained such as increase in hardness of material.

SMAT process was done in SMAT set up developed in COEP workshop. Surface treated specimens were characterized using Optical microscope, Scanning electron microscope, X-ray diffraction, Gamry instrument, Roughness testing machine and microhardness measurement. Results showed that there is increase in hardness value at surface and in cross section, also the transformation of phase from Austenite to martensite. X-ray diffractogram recorded from the surface of specimens of as received and SMATed shows there decrease in crystallite size of SMATed sample and increase in microstrain and dislocation density of SMATed specimen. The result obtained from Electron back scattered diffraction (EBSD) shows there formation of mechanical twins due to which there grain refinement occurs and nano grains are formed.

Mechanical alloying is possible with SMAT process. Copper powder embedded in aluminium surface with SMAT technique. Balls take powder with them and powder adheres on the surface and coated on the surface.

Through numerous experiments performed in the current project, and theoretical inputs, it was confirmed that SMAT is a successful method to improve surface properties.

Dissertation Report

on

"Conducting polyanilline on hot dip galvanized low carbon steel for corrosion protection"

Submitted by

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In partial fulfillment for the award of the degree

Of

M. TECH (PHYSICAL METALLURGY)

In

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

COLLEGE OF ENGINEERING, PUNE

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Under the guidance of

Dr. P. P. DESHPANDE

June 2014

Abstract

Conducting polyaniline based paint have been applied on hot dip galvanized low carbon steel samples. The duplex coatings of conducting polyaniline based paint and zinc coating on low carbon steel provided excellent synergetic effect. The corrosion protection aspects of conducting polyaniline based and red primer coatings, in both – intact and damaged form, on hot dip galvanized low carbon steel were investigated in aqueous 3.5% NaCl solution by potentiodynamic polarization studies, open circuit potential measurements, electrochemical impedance spectroscopy. Corrosion rate of conducting polyaniline based and red primer paint coating on hot dip galvanized low carbon steel in 3.5% NaCl is found to be 1.01mpy and 2.08mpy respectively which is about 9.1 and 4.6 times lower than that of unpainted low carbon steel respectively in same medium. The coating resistance of conducting polyaniline paint coated hot dip galvanized steel in intact and damaged condition just after immersion is 1120.3 Ω/cm^2 and 558.5 Ω/cm^2 respectively. After 24 hours this is decreased to 730.2 Ω/cm^2 and 439 Ω/cm^2 respectively. Then increased to 813.5 Ω/cm^2 to 310.2 Ω/cm^2 respectively after 72 hours. At the end of 96 hours the coating resistance of intact and damaged coating was decreased 446.6 Ω/cm^2 to 39.6 Ω/cm^2 respectively. Increment in the coating resistance of intact coating and damaged coating value indicates the dissolution of iron but subsequent possible formation of passive Fe_2O_3 layer this reveled that conducting polyaniline based paint on hot dip galvanized low carbon steel is better for long term corrosion protection even in intact and damaged condition. The duplex coating of conducting polyaniline based coating and zinc coating controls corrosion of low carbon steel in neutral medium by anodic protection.

A

Dissertation Report

On

Development of High Temperature Semiconductor β-$FeSi_2$ for Thermoelectric Application

Submitted in partial fulfilment of the requirements for the Degree of

Master of Technology in Metallurgical Engineering

By

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MIS No: - 121226015

M.Tech - Second Year

(Physical Metallurgy)

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2013-2014

ABSTRACT

Thermal management and energy crisis have been two major problems in this 21st century. Amongst the thermoelectric materials, β-$FeSi_2$ is emerging as an alternative promising high temperature semiconductor useful for temperature from 600-900°C.

Mechanical Alloying process has been used to produce iron-silicide. The powders of pure electrolytic iron (98.9%) and silicon (99.9%) were blended in a Attritor mill (AM) and Cryo mill (CM) for 4-8 h. As milled powders were of metastable state and fully transformed to β-$FeSi_2$ phase by subsequent isothermal sintering at 800°C. Then it was compacted at 700 MPa and subjected to different heat treatment cycles. Phase transition during the process was investigated using DTA, XRD, SEM and EDS.

As consolidated iron silicide consisted of untransformed α-$FeSi_2$ and ε-FeSi phases. Subsequent isothermal sintering at 800°C in vaccum led to thermoelectric β-$FeSi_2$ phase transformation. Fraction of β-$FeSi_2$ increased with sintering period as well as milling period.

Keywords: Particle Size, Phase Transformation, Density, Electrical Conductivity.

Dissertation Report

On

"SYNTHESIS AND CHARACTERIZATION OF FEW LAYER GRAPHENE COATED COPPER FOR CORROSION PROTECTION"

Submitted by

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In partial fulfillment for the award of the degree

of

M. Tech

(Specialization - Physical Metallurgy)

IN

DEPARTMENT OF METALLURGY AND MATERIALS SCIENCE

Under the guidance of

Prof. A.M.MORE **and** **Dr. SUNIL R. PATIL**

DEPARTMENT OF METALLURGY AND MATERIAL SCIENCE

COLLEGE OF ENGINEERING, PUNE.

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ABSTRACT

This work reports the successful synthesis of graphene using solid carbon sources like graphite powder and commercially available graphite pencil. After the synthesis heat treatment sample surfaces were cleaned using mechanical exfoliation and sonication method. Graphene synthesis using solid carbon sources is economical and technologically simple method as compared to chemical vapor deposition (CVD). Graphene was characterized using Raman spectroscopy before and after it is transferred to silicon wafer. The corrosion response of graphene coated copper substrate is also reported. The corrosion behavior of the bare copper and graphene coated samples is characterized by Cyclic Voltammetry, Potentiostatic polarization and Electrochemical Impedance Spectroscopy. This study claims successful synthesis of graphene on copper substrate and ~500% improvement in the corrosion resistance of copper.