**METALLURGICAL ENGINEERING**

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Author : **RAMAVATH BHEEKYA NAIK**

Title of the thesis : **DEVELOPMENT OF HIGH STRENGTH AND HIGH ELECTRICAL CONDUCTIVITY CU ALLOYS/ COMPOSITES THROUGH FRICTION STIR PROCESSING**

Guide : **Dr. R. AROCKIA KUMAR**

Degree : **Ph. D.**

Student ID No. : **714028**

**ABSTRACT**

 Copper (Cu) is a high electrical conductivity material with a wide range of applications from heavy-duty electrical contacts to resistance welding electrodes. Due to poor mechanical and wear properties it cannot be used in applications demanding high strength and high wear resistance. Therefore, the strength and wear resistance of the Cu needs to be enhanced without compromising the electrical conductivity. Methods such as alloying, precipitation hardening and dispersion strengthening (by reinforcing hard ceramic particles) have widely been used to enhance the strength of pure copper. However, in most of the cases it resulted in deteriorating the electrical conductivity. For real-world applications, the surface is only expected to have high hardness and wear resistance. Then, it is good enough to improve the properties of the surface. The present research work is therefore attempted to improve the surface properties of Cu based alloys/ composites through a solid-state process known as friction stir processing (FSP). The friction stir process is a modification of friction stir welding (FSW) process, has shown to be effective to enhance the surface properties through micro structural modification e.g. introduction of dislocations, refinement of grains and precipitates and incorporation of hard ceramic reinforcements.

 In the initial phase, the surface of the commercial pure copper was processed using single pass FSP at two rotational speeds (600 rpm and 900 rpm) and varying traverse speeds (50 to 200 mm/min steps of 50 mm/min) with an objective to improve the mechanical and wear properties without deteriorating the electrical conductivity. FSP facilitated improved hardness (58%) and wear resistance at the 600 rpm-200 mm/min. However, a slight deterioration of the electrical conductivity (1.8%IACS) was observed which is negligible.

 In the second phase, the surface of commercially available copper alloys such as Cu-Zr, Cu-Cr, and Cu-Zr-Cr were processed using fixing the tool rotational speed 600 rpm and varying the traverse speeds from 50 to 200 mm/min in steps of 50 mm/min. The hardness of the alloys (Cu-Zr: 41%, Cu-Cr: 33%, and Cu-Cr-Zr: 33%) was improved and the wear resistance enhanced after FSP. Once again, there was an insignificant reduction in electrical conductivity

In the third phase, Copper-Tungsten (Cu-W) and Copper-Graphene (Cu-Gr) composites were prepared by single pass FSP for fabricating high strength and high electrical conductivity copper surface composites. The Cu-W composite was prepared by varying the traverse speed (50 to 200 mm/min steps of 50 mm/min) at a constant rotation speed of 600/900 rpm. The hardness of Cu-W composite was increased up to 130% (600 rpm and 50 mm/min). The hardness of Cu-Gr composite was increased up to 64% (900 rpm and 200 mm/min). The wear rate of the composites decreased without much deterioration (Cu-W: 1.8%IACS and Cu-Gr: 1.4% IACS) in the electrical conductivity.

 Overall, the work suggests the single pass FSP has improved the mechanical and tribological properties without deteriorating the electrical conductivity for pure copper, copper alloys (Cu-Cr-Zr, Cu-Cr, and Cu-Zr), Copper-Tungsten (Cu-W) and Copper-Graphene (Cu-Gr) composites.

**METALLURGICAL ENGINEERING**

Author : **PAPIYA BISWAS**

Title of the thesis : **PROCESSING OF TRANSPARENT MAGNESIUM ALUMINATE SPINEL USING DIFFERENT TECHNIQUES AND EVALUATION OF ITS PROPERTIES**

Guide : **Dr. M. K. MOHAN**

Degree : **Ph. D.**

Student ID No. : **701429**

**ABSTRACT**

 Transparent magnesium aluminate (MgAl2O4) spine is a material of special interest due to its optical properties coupled with excellent mechanical properties especially for strategic applications. In view of this, spinel is proposed as a futuristic material in addition to the existing demand in the country which has lead to significant scientific and technological importance. Prime motivation of this research is to modify or develop new processes for shaping, densification and to achieve optical, thermal and mechanical properties near theoretical values.

 Accordingly, in the current study a simple and effective thermal passivation process has been developed to address the issue of inherent hydrolysis associated with the basic nature of spinel while shaping through aqueous based process. Transparent spinel ceramics with a density of 3.58 g/cm3, transmission close to 85% in 3-5 micron region and mechanical properties (flexural strength – 210 MPa and hardness – 1300 kg/mm2) at par with literature values was successfully fabricated through defect free slip casting process followed by pressure less sintering and hot isostatic pressing. Additionally, efforts were also made to synthesis in-house spinel powder targeting benchmark specification of imported powder

 Attempts were made to fabricate magnesium aluminate based cellular ceramics such as honeycombs through extrusion processing and foams through polymeric sponge processing. Thin walled dense honeycomb with channel density of 900 CPSI and relative density of 0.45 has been developed in the present study. An environmental friendly process on methyl cellulose based gelcasting technique is developed and employed for the first time to fabricate transparent honeycomb structure. Transparent spinel blanks and foams are explored for biomedical applications as orthodontic brackets and scaffolds. Feasibility of fabrication of magnesium aluminate spinel through chemical vapour deposition technique has been established through a thermodynamically favourable reaction-2AlCl3(g) +MgCl2(g)+4H2(g)+4CO2(g)+Heat=MgAl2O4(s)+8HCl(g)+4CO(g) .

 A new process of encapsulating the powder in a platinum foil and obtaining carbon contamination free samples without any discoloration has been demonstrated in this study. This provides a single step process which can be extended to other ceramics as well. Additionally flash sintering of magnesium aluminate spinel has been carried out in collaboration with National Institute of Materials Science (NIMS), Japan and occurrence of flash is established (1000 V/cm at 1410oC) resulting in densification of sample (>98% TD) theoretical density. Further, the samples upon hot isostatic pressing achieved close to theoretical transmission of 81%.

**METALLURGICAL ENGINEERING**

Author : **PRAKASH**

Title of the thesis : **INFLUENCE OF THERMOMECHANICAL TREATMENT ON MICROSTRUCTURE, MECHANICAL AND CREEP RUPTURE BEHAVIOUR OF REDUCED ACTIVATION FERRITIC- MARTENSITIC STEEL**

Guide : **Dr. G. V. S. Nageswara Rao**

Degree : **Ph. D.**

Student ID No. : 715053

**ABSTRACT**

In the present investigation, the effect of thermomechanical treatment (TMT) on microstructure, hardness, tensile properties, flow and work-hardening behaviour, creep deformation and rupture behaviour of 9Cr-1W-TaV reduced activation ferritic martensitic (RAFM) steel has been studied and compared with those of the conventional normalised and tempered (N+T) RAFM steel. The TMT was carried out by performing warm rolling the steel in the austenitic phase field at 973 K before the martensitic transformation on cooling followed by ageing at warm rolling temperature and then tempered at 1038 K for 90 min. A limited number of trials were also carried out on TMT of the steel in the ferritic phase field and its effect on tensile and creep deformation for comparison purpose at some instances. Upon TMT, the tempered martensitic structure of the steel underwent refinement in terms of lath size and large quantity of finer M23C6 and MX precipitates with an increase in dislocation density than those in the N+T steel. TMT of the steel increased its hardness compared to the N+T condition. Tensile strength of the steel increased on TMT over the investigated temperature range of 300-923 K and at a strain rate of 3 × 10-4 s-1. The increase in strength was accompanied without appreciable loss of ductility, perhaps with increase in ductility. The tensile flow behaviour of the steel has been assessed in the frame work of Voce’s constitutive equation. The strength related parameters of the constitutive equation, i.e. initial stress and saturation stress increased on TMT, while the recovery related parameter (*nV*) decreased marginally. The enhancement of tensile strength and ductility of the steel on TMT is considered due to microstructural refinement and its enhanced stability as reflected in appreciable lower relaxation and creep rates and the delayed onset of tertiary stage of creep deformation. The plots of work hardening rate () vs. stress () revealed transient stage and stage-III of work hardening for both TMT and N+T steels. However, the TMT processing increased the work hardening of the steel, as manifested by the shift in  vs.  plots to higher stresses at all the temperatures and an increase in the fraction of transient stage of  vs.  plot up to a temperature of 773 K. Further, an increase in stress to onset of stage-III and the decrease in rate of dynamic recovery (Stage-III) have been observed for TMT steel, in comparison to the steel in N+T condition. This is attributed to the decrease in inter-barrier spacing of the obstacles in TMT steel measured analytically. The above-mentioned observations substantiated the beneficial effect of TMT on tensile flow and work hardening of the RAFM steel.

 Creep tests were carried out at 823 K over the stress ranges 180-300 MPa. TMT processing of the steel decreased the minimum creep rate ( min  ) with corresponding increase in time to onset of tertiary stage of creep deformation, rupture life (tr) and creep rupture ductility ( f  ). The stress exponent value (n), obtained from minimum creep rate vs. stress plot, increases upon TMT processing, indicating high resistance to creep deformation than in the N+T steel. Resisting stress as estimated based on the Lagneborg and Bergman method is found to increase on TMT processing and is associated with high damage tolerance parameter, defined as   f (min .tr ) . Enhanced creep deformation and rupture strength of the TMT steel, compared to N+T steel, is attributed to the microstructural refinement. Post-creep microstructural investigations show higher microstructural stability of the steel on TMT processing and are in line with the observed high damage tolerance parameter (), longer time to onset of tertiary creep and rupture life. Keywords: Reduced activation ferritic-martensitic steel; Thermomechanical treatment; Microstructural stability; Tensile properties; Constitutive equations; Work-hardening behaviour; Creep deformation; Creep damage tolerance parameter.

**METALLURGICAL ENGINEERING**

Author : **POTHURI RAMBABU**

Title of the thesis : **TENSILE, MODE I AND MIXED MODE I/III FRACTURE BEHAVIOUR OF AEROSPACE GRADE ALUMINIUM**

 **ALLOY AA 2219 FORGINGS**

Guide : **Dr. M. K. Mohan**

Degree : **Ph. D.**

Student ID No. : **701148**

**ABSTRACT**

The aim of the aircraft and missile designers is to keep the weight at minimum to enhance the fuel efficiency and at the same time, improve the overall performance in terms of capability. The attractiveness of aluminium is that it is a relatively light weight metal with low cost, good fabricability and well established in terms of NDT and Inspection routines/ procedures. Further, aluminium alloys can be heat-treated to fairly high strength levels; and it is one of the most versatile high performance materials, which usually correlates directly with lower costs. There is particular interest in these alloys for application in critical structural components in aircraft and missile systems which require specified/select combinations of mechanical properties for overall improved working efficiency.

 The strength of many aluminium alloys is increased with the heat treatment called precipitation hardening. Among the 2XXX series aluminium alloys, alloy AA 2219 has favourable weldability characteristics coupled with higher usage temperature thereby finding many structural applications for this alloy in aerospace industry. Increasing application of aluminium alloys in different operating conditions necessitated change in design criteria of structures. While designing the structures, yield strength and fatigue strength are considered important for resisting deformation and failure under service loads. However, in the presence of cracks and other flaws, it is the fracture toughness that invariably becomes the most important parameter. Minimum fracture toughness requirements have become more stringent, and in the high-strength alloys the inverse relationship between strength and toughness, limits the level of yield strength that can be safely employed by the designer. The latest philosophy in designing the structures is with the assumption that cracks are generally exist in an engineering structure. The study of fracture behaviour is useful and perhaps even essential in determining whether these cracks will grow large enough to cause the failure before they are sure to be detected during a periodic inspection. The focus in this design approach is on using materials with high fracture toughness and those which exhibit slow crack growth.

 Mode I fracture toughness tests were conducted generally to know the fracture behaviour of materials. However, assessment of structural integrity of structures requires investigating the combination of tensile (mode I) and shearing (mode II) or tearing (mode III) loadings. This reason makes it necessary to the study of fracture resistance under such mixed-mode conditions. Therefore, the present study is conducted to evaluate the effect of mode III component on mode I in mixed mode I/III fracture toughness. Experiments were conducted in different ageing conditions of the alloy AA 2219 forging and apart from tensile and mode I fracture toughness, mixed mode I/III fracture toughness tests were performed with different loading angles.

 The thesis is arranged in seven different chapters. Chapter 1 reports on the role of aluminium alloys in aerospace industry and importance of mixed mode fracture toughness testing. In Chapter 2, extensive literature review has been made on various thesis aspects, emphasizing the testing of mixed mode fracture toughness along with procedures adopted for evaluation. A critical review of the available literature on mixed mode fracture toughness was also presented in order to identify the gaps left which became the objectives of the present study. Chapter 3 discusses about the material, properties evaluation techniques and characterization methods used for present investigation. Chapter 4 presents comprehensively all the results obtained from the present study and Chapter 5 is devoted to include detailed discussion to explain the results in a more elaborate and systematic way. Chapter 6 summarises the studies conducted and also, presented the overall conclusions that emerge from this thesis work. Chapter 7 shows the future scope of work.

 In the present study, mode-I fracture behaviour and influence of externally imposed mode III (out of plane shear and tearing) components on the mode-I fracture behaviour in aluminium alloy AA 2219 forgings have been analysed and discussed in four different ageing conditions. The measured fracture toughness in L-T orientation of the alloy in peak aged condition is the lowest (26.8MPa√m) among all the 4 ageing conditions; whereas, 31.5MPa√m is highest in L-T orientation in over aged condition. Alloy in T-L orientation has shown lowest fracture toughness of 21.4MPa√m in the peak aged condition and 28.8MPa√m is highest in over aged condition. This trend indicates the conventional inverse relation with fracture toughness decreasing with increasing yield strength. In L-T orientation in the peak aged condition only valid KIc has been observed whereas in all the other three conditions in which some shear lips were observed indicating invalidity in thickness due to prevailing of plane stress conditions and this shows the ability of the material to accommodate large strains due to higher „n‟ value. The detailed analysis of the data and the SEM fractographic observations confirm that the controlling fracture mechanism in-deed is the high energy ductile dimple fracture. Identical trends are observed in both L-T and T-L orientations and it can be concluded that there is no much effect of directionality in the fracture properties of this alloy in mode-I fracture toughness testing. During mixed-mode I/III fracture toughness testing there is an increase in total fracture toughness observed in all the tests related to different loading angles. The Ktq obtained is more than KIc in both L-T and T-L directions in all the four ageing conditions viz. LUA, UA, PA & OA with different loading angles viz. 30°, 45° and 60°. Similar trend is observed in both L-T and T-L directions and at any test condition mode-I fracture toughness is the lowest. Therefore, it is safe to design this alloy based on mode-I fracture toughness in aerospace applications. When the component is expected to function under shear load, design calculation based on Mode I fracture toughness results in higher safety factor. However, for military and combat applications where lower safety factors are practiced to keep the weight at lowest possible mark, the design of various components of alloy AA 2219 should be based on the actual (even conditional) fracture toughness values – the total fracture toughness, Ktq.

**METALLURGICAL ENGINEERING**

Author : **U. RAVI KIRAN**

Title of the thesis : **EFFECT OF PROCESSING AND ALLOY CHEMISTRY ON MECHANICAL PROPERTIES OF TUNGSTEN HEAVY ALLOYS**

Guide : **Dr. G. V. S. NAGESWARA RAO**

Degree : **Ph. D.**

Student ID No. : **714144**

**ABSTRACT**

 Effects of heat treatment and alloying on microstructure and mechanical behavior of tungsten heavy alloys have been studied. An attempt has been made to carry out a systematic correlation between key microstructural parameters such as W-particle size, W-W contiguity, matrix volume fraction and chemistry of different phases and mechanical properties such as tensile strength, % elongation to failure and impact toughness.

 The effect of cyclic treatment on mechanical properties of 90W-6Ni-2Fe-0.5Co- 1.5Mo was investigated in both as heat treated and swaged conditions. Incorporation of cyclic heat treatment resulted in improvement of both impact and fracture toughness. These enhancements were also retained in swaged condition. This was attributed to penetration of the matrix phase thereby eliminating in part W-W boundaries. Improved properties were also associated with reduced W-W decohesion as observed in the fractograph of the impact and fracture toughness specimens. An explanation for the matrix penetration was provided and corroborating microstructural evidences that is ingress of the matrix phase between the W particles with simultaneous reduction in the contiguity vales were given. This study brought out the importance of cyclic treatment in the enhancement of mechanical properties of heavy alloys.

 Three alloys based on 93% W, namely WNF (93W-4.9Ni-2.1Fe), WNC (93W- 4.2Ni-1.2Fe-1.6Co) and WNR (93W-4.9Ni-1.9Fe-0.2Re) were evaluated for microstructure, tensile and impact in heat treated and swaged condition in order to study the effect of Co and Re. The addition of Co resulted in improvement % elongation and impact. This was attributed to increased solubility of W in the matrix phase that leads to increased volume fraction of the softer matrix and decreased contiguity. These were further corroborated by fractographic evidences that showed reduced W-W decohesion and the presence of dimples corresponding to matrix failure. Re addition on the other hand increased strength at the expense of elongation. Increased strength was explained in terms of reduced mean free path in the matrix phase, a result of finer tungsten grain size. Following swaging while the tensile strength increased, % elongation came down. Work hardening analysis showed that all alloys in heat treatment condition showed higher work hardening exponent as compared to those in heat treated plus swaged condition. Rationale for this behavior was provided.

 Finally the effect of high energy milling on microstructure and mechanical properties were investigated in alloys containing 90%. Attempt was especially made to look for undissolved Re particles in conventionally processed material and explore if it was possible eliminate these particles by including an additional step of high energy milling. Mechanically alloyed W-Re solid solution is formed following high energy milling. Subsequent liquid phase sintering leads to finer microstructure due to more efficient dissolution of Re in the matrix (prior liquid) phase that is confirmed by the presence of undissolved Re rich particles in the conventionally sintered alloy that has not employed high energy milling of W- Re and higher Re content in the matrix phase that used mechanically alloyed W-Re for LPS. As a result, the properties of the alloy processed with MA W-Re powder were superior to the one that was conventionally processed. The presence of finer W-grains was explained in terms of Re rich layer around W particles that reduced the diffusion kinetics.

 In brief this thesis explores the possibilities of property enhancement in tungsten heavy alloys by incorporating some novel processes such as cyclic heat treatment and mechanical alloying. Attempt is also made to understand the trends in properties in terms of underlying microstructure and key microstructural parameters that govern the properties are clearly brought out.

**METALLURGICAL ENGINEERING**

Author : **VIBHUTI ROSHAN**

Title of the thesis : **EVELOPMENT OF PROCESS FOR UTILIZATION OF IRON ORE SLIME OF BAILADILA REGION THROUGH HYBRID PELLET SINTERING**

Guide : **Dr. G. V. S. NAGESWARA RAO**

Degree : **Ph. D.**

Student ID No. : **701215**

**ABSTRACT**

 In Bailadila region of India, about 8-10% of slimes are being generated during mining and processing of iron ore. These slimes contain valuable iron source. However, these slimes are ultrafine in size and not suitable for subsequent utilization in iron making and steel making processes. Hence, these slimes are rejected and stored in tailing dams. This result in dual impact on mining operations: firstly, the loss of valuable iron value as waste and secondly, creating space constraint as huge quantities of slime are being accumulated in tailing dams. With a view to find out avenues for utilization of iron ore slimes, a systematic beneficiation studies were undertaken with slime sample from deposit-5 of Bailadila region. In the present work, iron ore slimes from Bailadila mines located in Chhattisgarh state were selected for the study. The iron ore slimes were subjected to various standard procedures for studying their initial physical and chemical properties. The slimes were also studied microscopically by using optical and scanning electron microscopes to establish the status of liberated minerals. Based on these characterization inputs, gravity and wet magnetic techniques were used individually to beneficiate the iron ore slimes. Dry magnetic separation process was also tried considering water scarcity in future near mine site. However the process was plagued by high dust generation and relatively low recovery and productivity. As the individual techniques have yielded a limited success, combination of gravity and magnetic techniques were used to obtain beneficiated slime with maximum recovery of iron value (more than 64% Fe content). At laboratory scale, combination of hydro-cyclone followed by two stage wet high intensity magnetic separation (WHIMS) reveal that the concentrate assaying 64.74% Fe(T), 2.39 % SiO2 and 1.82% Al2O3 with a recovery of 85.16% Fe value could be produced. Based on the flow sheet developed at laboratory scale, a pilot plant was also designed for the beneficiation of iron ore slime on large scale. The laboratory scale results were validated by pilot scale beneficiation facility. The final flow sheet of beneficiation of slime was processed on pilot scale at 1 T/h capacity plant. The pilot scale studies reveal further improvement in the grade of the concentrate and the recovery of Fe value. Iron ore concentrate of 65.71 % Fe(T), 2.18% SiO2 and 1.64% Al2O3 with 87.31 % recovery is generated in pilot scale test. The concentrate thus obtained from pilot plant was used for the manufacture of micro-pellets of various sizes (between 1 and 10 mm) of sufficient strength under optimized conditions in disc pelletizer. The green properties of micro-pellets were optimized with respect to binder content, moisture and surface area. The micro-pellets prepared from slime concentrate obtained after beneficiation of Bailadila deposit-5 slime has exhibited excellent physical properties at 0.5% bentonite content and 9.25 % moisture at surface area of 1780 cm /gm. The micro-pellets thus obtained were used in hybrid pellet sinter making in pot grate sinter furnace. The sinter produced exclusively from micro-pellets exhibited better bed permeability and void fraction compared to conventional sinter. The results confirm that the sinter prepared from micro-pellets of size 3-6 mm can effectively replace iron ore fines in sinter making. During hybrid pellet sinter making, parameters such as moisture content, coke content in the sinter mix, bed height and ignition time for making sinter were studied and optimized. The optimized conditions for making hybrid pellet sinter are found to be: 6.5% moisture, 5.5% coke breeze, 350 mm bed height and 120 s ignition time. Six batches of sinter with basicity ranging from 0.5 to 3.0 were produced from micro-pellets of size 3-6 mm and the effect of basicity on properties of hybrid pellet sinter was also studied. The sinter thus produced from micro-pellets was subsequently evaluated for its performance under blast furnace conditions. The performance of hybrid pellet sinter under blast furnace conditions is observed to be better in comparison to conventional sinter with respect to reduction and strength at high temperature under reducing condition. The studies reveal that the metallurgical properties of the sinter (with basicity 2.5) are observed to be comparatively high with high relative reducibility of 65.7%. The higher reducibility of sinter in turn promotes higher indirect reduction and as a result significant reduction in coke rate can be obtained. On the whole, the iron ore slime from Bailabila (deposit-5) mines is convertible into useful blast furnace feed for iron making, there by alleviating the problem of space for safe disposal of slime as waste. Keywords: Iron ore slimes; gravity and wet magnetic separation; floatation; micro-pellet making; hybrid pellet sinter; blast furnace simulation.

**METALLURGICAL ENGINEERING**

Author : **KAKU SAI MAHESH YADAV**

Title of the thesis : **PRODUCTION AND PROPERTIES EVALUATION OF ZRB2 REINFORCED AL/AL ALLOY COMPOSITES**

Guide : **Dr. ASIT KUMAR KHANRA**

Degree : **Ph. D.**

Student ID No. : **714030**

**ABSTRACT**

This thesis makes an attempt at designing of aluminium (Al) - zirconium diboride (ZrB2) composite produced through powder metallurgy (PM) technique. Pressureless sintering technique was used in order to produce samples for the present research work. Optimization of ZrB2 reinforcement in the Al matrix was performed by analyzing the structure-property correlation study and corrosion behaviour.

 Al matrix was alloyed with 4wt% copper (Cu) and varying amounts (0-1 wt%) of magnesium (Mg) as alloying elements. The densification behavior of the alloy preforms were analyzed and optimum alloying conditions were drawn from the analysis. Combining optimum conditions of both the systems Al-ZrB2 and Al-Cu-Mg, were designed with enhanced properties. Comparative studies were carried out between the optimum conditions of both systems with alloy/ composite. An attempt was also made to produce the composites by other technique such as vacuum arc melting (VAM).

 The thesis has five chapters. The introduction (chapter-1) section deals with the evolution of Al and Al alloys with wide range of reinforcements in order to produce composites. Literature review (chapter-2) consists of brief explanation of the Al alloys and its composites, which are produced by various routes. Deformation behavior of Al composites are performed under various conditions by using uniaxial compression press. The effect of densification and deformation on the hardness and corrosion of the composites is presented.

The third chapter consists of the experimental procedure involved in the production of composites through pressureless sintering, deformation and corrosion studies. The characterization techniques used in the present investigation have been explained briefly in the same chapter. Chapter-4 deals with the different results obtained through experimentation and thorough discussion of all the material systems involved in the design of alloy composites. Finally in chapter-5, the conclusions are drawn based on the results and discussion.

 Borides are ultra-high temperature ceramic (UHTC) materials used in wide range of applications. Borides have properties like high chemical stability, high hardness and good thermo-electric properties. Zirconium diboride (ZrB2) was such significant boride ceramic which exhibit superior

properties like high melting point, extremely high hardness and wear resistance, low specific gravity, magnetic, electrical properties, with high mechanical properties and chemical inertness at elevated temperatures. The ZrB2 based materials exhibit great dimensional stability which was of prominent importance in automobile functioning. The ZrB2 powder can be used in order to manufacture ceramic reinforced composites. The present study deals with the production of aluminium based metal matrix composites (MMC) by powder metallurgical route. Different composites are made by adding different amounts of ZrB2 (0, 2, 4 and 6wt %) of three aspect ratios (ASPR) of 0.35, 0.5 and 0.65 respectively. The powder mixture is compacted and pressureless sintered at 550oC for 1 hour in control atmosphere (Argon gas). The relative density of the sintered preforms is found to be 90% approximately. The hardness of the composites increased with the amount of ZrB2 in the composite. Sintered preforms were used as workpiece materials for deformation study. Deformation study was performed at different temperatures in order to find the effect of temperature on the densification behaviour and other mechanical properties. Densification improved with the extent of deformation up to failure. Densification decreased with the increase in ASPR. The hardness of the deformed composite increased with the extent of densification. Potentio-dynamic polarization studies were performed on the deformed performs to find the effect of mechanical working. The 3.5% aqueous NaCl solution was used as electrolyte. The corrosion rate was found to decrease with the extent of deformation whereas reverse trend was noticed with the increase of ZrB2 particles in the composite.

 Alloying of monolithic metal powders plays a significant role in the enhancement of properties. Aluminium powder matrix is alloyed with copper and magnesium through powder metallurgy route. By maintaining 4wt% Cu as constant, the different wt. % (0.25, 0.5, 0.75 and 1) of Mg are mixed in matrix to produce different powder alloy performs. The powder performs are triaxially compacted in a tool steel die and sintered at 550oC for one hour in high pure argon gas atmosphere. The sintered alloy performs are subjected to uniaxial deformation at three different temperatures, (25, 400 and 500oC). The presence of secondary phase particles CuAl2 appeared after sintering of green compacts which were analyzed through X-ray diffraction (XRD). Hardness of the sintered composites increased with the increase in the amount of Mg in the alloy perform. Al-4Cu-0.5Mg alloy performs were highly densified. The Relative density of alloy performs decreased with Mg in excess of 0.5wt%. Hardness and the relative density increased with the extent of deformation. Potentio-dynamic polarization was performed on deformed. performs to evaluate the effect of deformation. Corrosion rates and hardness of the alloy preforms were correlated with the relative densities for evaluating the effect of the extent of deformation and compositional variations.

 Considering the optimum compositions obtained from Al-ZrB2 system and Al-Cu-Mg system comparative studies were done between Al, Al-2ZrB2, Al-4Cu, Al-4Cu-0.5Mg, Al-4Cu-2ZrB2 and Al-4Cu-0.5Mg-2ZrB2. Densification improved with the extent of deformation. Out of all compositions Al-4Cu-0.5Mg was highly densified. With the combination of optimum compositional conditions in both systems Al-4Cu-0.5Mg-2ZrB2 alloy composite was produced and tested. The alloy composite was measured to have high hardness and optimum relative density. Corrosion rate was slightly higher than other composites which is at the cost of hardness and relative density.

The Al and Al- alloy composites which were compared earlier were also produced through vacuum arc melting (VAM). The results of the VAM alloy composites were compared with the alloy composites produced through PM route. Hardness of the alloy composite produced through VAM were measured to be higher than composite produced through PM technique.

**METALLURGICAL ENGINEERING**

Author : **UMME THAHIRA KHATOON**

Title of the thesis : **NOVEL STRATEGY TO SYNTHESIZE METAL AND METAL**

 **OXIDE NANOPARTICLES FOR ANTIMICROBIAL AND**

 **ANTICANCER APPLICATIONS**

Guide : **Dr. G. V. S. NAGESWARA RAO**

Degree : **Ph. D.**

Student ID No. : **701312**

**ABSTRACT**

 Microorganisms are omnipresent in many regions of our planet, and their presence affects the surroundings wherever they grow. The influence of microorganisms on their surroundings is often sensible or harmful with respect to the human life. Therefore, the management of harmful effect of microorganisms is very important issue. To manage this problem, medical practitioners have suggested many techniques and medicines with multi or broad spectrum activity equivalent to antibiotic drug, kanamyc in, and penicillin respectively to overcome infections caused by microorganisms. However, frequent treatment by those medications makes many microorganisms to become resistant towards these medications. Therefore, new classes of antimicrobial drugs based on application of nanomaterials become very important target of research. Recently robust antimicrobial activity has been determined for many nano materials. Some metal and metal oxides based nanoparticles such as silver (Ag), silver oxide (AgO), gold (Au), titanium dioxide (TiO2), calcium oxide (CaO), copper oxide (CuO), silica (SiO2) and magnesium oxide (MgO) have been reported to show antimicrobial activity. Because of high surface to volume ratio, metal and metal oxide nanoparticles exhibit different and/or advanced chemical properties over bulk material. These chemical properties of nanoparticles are influenced by their size, shape, crystal planes and other physical characteristics.

 **The Objectives of the present investigation are:** Synthesizing pure Ag, AgO and CuO NPs without using stabilizing and capping agents at six different concentrations with various reducing agents. Designing a single experimental procedure to synthesize Ag, AgO and CuO NPs. Optimizing the reducing agent for proper yield. Optimizing the concentration of reducing agent. Optimizing the concentration and reducing agent with respect to biomedical applications (anti microbial and anti cancer studies). Studying the effect of change of concentration on particle size, zeta charge, morphology, on biomedical applications.

**The thesis is arranged in seven different chapters.**

***Chapter 1*** explains the introduction about nanotechnology, nanomaterials synthesis methodologies and their applications. Depending on that, list of objectives have been designed to fill the gaps and demands of nanotechnology.

In ***Chapter 2,*** extensive literature review has been presented to synthesize of metal nanoparticles from various techniques and methodologies. This chapter also critically reviews the synthesis methodology of metal nanoparticles which are more stable and considered for biomedical applications. Different synthesis strategies have been explained in this chapter.

***Chapter 3*** discuss materials, methods and characterization techniques used to investigate the properties of metal nanoparticles. Also, a brief explanation about antimicrobial and anticancer studies has also been presented.

***Chapter 4*** presents the synthesis, characterization of silver nanoparticles (Ag NPs) synthesized by two different reducing agents with respect to different molar concentrations of precursors (0.02 M, 0.04M, 0.06 M, 0.08 M, 0.1 M, 1.0 M) and their antimicrobial and anticancer studies. This chapter summarizes: simple and effective method of synthesis of Ag NPs from NaBH4 and C76H52O42 with well-defined size and antimicrobial and anticancer activity. To the knowledge of the author, this protocol has not been used by any researcher so far where no stabilizing or capping agents are used to synthesize pure Ag NPs. The X-ray diffraction (XRD) pattern on Ag NPs synthesized from both the reducing agents is in good agreement with standard JCPDS file reported by many researchers. The XRD pattern of the samples revealed the crystalline (FCC) lattice structure of elemental silver. Transmisson electron microsocope (TEM) have shown the average particle size in the range of 20-50 nm. Ultra violet visible (UV-visible) spectra analysis proved the presence of crystalline silver element and spectrum at 398-440 nm indicating the existence of Ag NPs. Dynamic light scattering (DLS) results revealed that the mean particle size range in between 30-70 nm and obtained Ag NPs are stable which is evident from TEM micrographs. Nanoparticles tend to effect the growth of microbes due to spherical shape and uniform morphology. Finally, this study has shown that silver nanoparticles have excellent antifungal activity against *E.coli* followed by *B.subtilis, candida albicans* and cancer cell lines namely, *MCF7, PVA1 and CACO2.* The Ag NPs synthesized from two reducing agents NaBH4 and C76H52O42 produced pure Ag NPs with spherical shape nanoparticles with pure crystalline FCC structure. On comparision, NaBH4 is more stable than C76H52O42. The stability of particles can be observed on UV-vis spectra, zeta potential analysis and TEM micrographs. Hence, NaBH4 products are more stable and dispersed compared to C76H52O42. The purity of Ag NPs are the same from XRD pattern the peaks are sharp. The yield of Ag NPs synthesized by NaBH4 is low compared to Ag NPs synthesized from C76H52O42.

***Chapter 5*** presents the synthesis, characterization of silver oxide nanoparticles (AgO NPs) respectively synthesized by two different reducing agents and their antimicrobial and anticancer studies. This chapter summarizes: Comparative study of AgO NPs synthesized using C6H5Na3O7 and C2H2O4 with respect to different molar concentrations of precursors (0.02 M, 0.04M, 0.06 M, 0.08 M, 0.10 M, and 1.00 M). During experiment and at the end of the experiment, no precipitates were formed or no deposit of silver was observed on the sides of beaker. Dark brown/ grey colour powder was formed. The transparent colourless solution was converted to the characteristic brown and white color when C6H5Na3O7 and C2H2O4 were used as reducing agent respectively. The occurrence of colour has indicated the formation of silver oxide nanoparticles. Yield has increased with increase in concentrations of precursors from 0.02M to 1.0 M. Mean particle size of AgO NPs is increased with increase in concentration. The agglomeration of particles is feasible and evident with TEM micrographs. There is no change in purity of particles with increase in concentrations as noticed via XRD diffractograms. The stability of particles is evident from studies on zeta potential charge. The higher the negative charge the lower the stability. The powder of AgO was formed which settled down when allowed to stand still for 30 minutes. It can be concluded that the reducing agent used was suitable to produce AgO NPs. AgO NPs have also shown good antimicrobial and anticancer activity against *E.Coli*, *B. Subtilis, Candida Albicans* and cancer cell lines namely, *MCF7, PVA1 and CACO2.*.

***Chapter 6*** presents the synthesis, characterization of copper oxide nanoparticles (CuO NPs) synthesized by three different reducing agents and their antimicrobial studies against five different bacterial strains. This chapter summarizes as, CuO NPs were synthesized without using capping and stabilizing agents. Here, CuO NPs does have ability to inhibit the growth of infectious bacteria and organism. On comparison, CuO NPs synthesized from NaBH4 has high inhibition activity followed by C2H2O4 and C6H5Na3O7. The reason is shape of NPs and which is circular followed by ellipsoidal and tube like structure. CuO NPs have high inhibition against *B. Subtilis, P.Putida, M. Luteus,* followed by *L. Lactis, E.coli.* CuO NPs have the capability to inhibit the growth of pathogens (disease causing micro organisms) and living organisms responsible.

***Chapter 7*** summarizes the conclusions drawn from the present study and also future scope of work. In this work, synthesis of Ag, AgO and CuO nanoparticles has been attempted through reducing silver nitrate and copper nitrate respectively. The novelty of the present work has been that no other supporting agents like capping and stabilizing agents other than reducing agents were used to reduce metal precursor. These nanoparticles were synthesized at 6 different molar concentrations. The observations made are, with increase in molar concentrations the size and shape of NPs are also increased. The change in molar concentrations of nanoparticles has shown change in the inhibiting the growth of microbes. The Zone of Inhibition (ZoI) of Ag, AgO and CuO NPs at lower molar concentrations has shown higher ZoI due to less size. Also, CuO NPs have shown more inhibition growth against microbes followed by AgO and Ag. The two main targets for futuristic use of these nanoparticles are: The synthesized product can be commercially used in market for biomedical applications or other applications, and the same process can also be studied on group 11 elements.

**Chapter 8** shows a comprehensive list of references which were used as basis for the present work followed by publications based on present study.