ADVANCED MATERIALS FOR EMERGING APPLICATIONS

INNOVATIONS, IMPROVEMENTS, INCLUSION AND IMPACT

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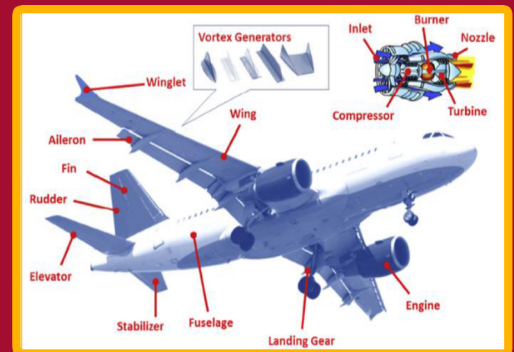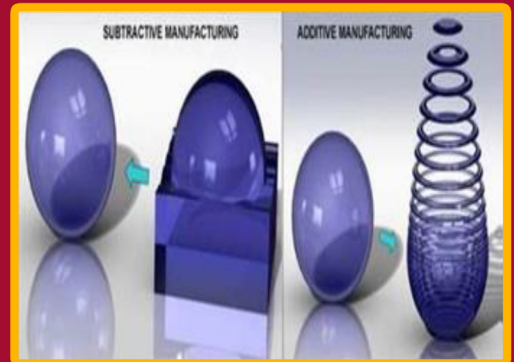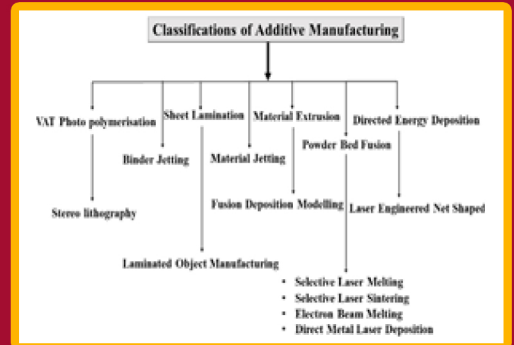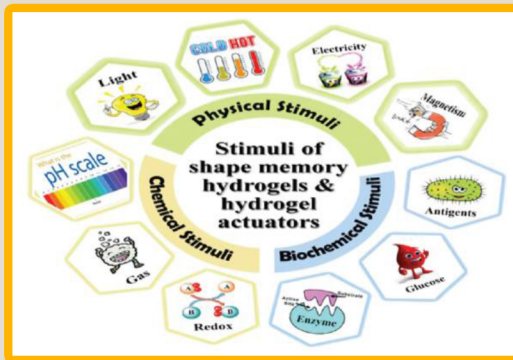**Manoj Gupta**

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PREFACE

"***Advanced Materials***" can safely be ascribed to those materials that have the potential of offering a useful combination of properties including physical properties, mechanical properties, electrical properties and chemical properties, which make them a potentially viable candidate for selection and use in a spectrum of applications spanning both performance-critical and non-performance-critical. This can be made possible through a healthy synergism of changes in composition, and changes in constituents coupled with the development and implementation of specialized and innovative techniques specific to both processing and synthesis. These materials have gradually grown both in stature and strength and include the following: (i) high value-added metals and their alloy counterparts, (ii) biomaterials, (iii) ceramics, (iv) ceramic-matrix composites, (v) electronic materials, (vi) high entropy alloys, (vii) multi-principal element alloys, (viii) metal-matrix composite materials, (ix) nanomaterials, (x) polymers, (xi) polymer-matrix composites, and (xii) semiconductors. The "emerging" materials and their traditional counterparts tend to differ significantly in terms of mechanical properties, physical properties and chemical properties. The properties offered by the newly developed and/or "emerging" materials can be customized, or tailored, specific to the primary purpose for use and application. Further, production and eventual commercialization of the family of "emerging" materials often tend to differ from the traditional counterparts in terms of the following input(s):

1. Overall importance of the engineered product(s).
2. The importance given to the different steps and related intricacies in the processing sequence (including fabrication), and
3. The economics specific to cost, based entirely on the scale of production.

The potential for an observable change in characteristics of the "emerging" materials and the markets to which they can serve are rapidly gaining in strength, which is made possible through a healthy combination of radically different materials and processes. Both cost benefits and structural advantage over the life of a newly developed material, or "emerging" material, can differ significantly from that of the traditional counterpart, thereby providing a clear indication that the traditional approaches to economic assessment may not be suitable and applicable to the family of "emerging" materials.

There does exist a need to establish meaningful boundaries for an "emerging" material in terms of both the input material and the end-product of interest, *i.e.*,

1. When in the processing chain or processing sequence can a material be classified as new, novel and "emerging" ?, and
2. When can an "emerging" material be chosen for use for a specific product?

In several cases, the categories of information that is both needed and essential with specific reference to an "emerging" material is the same as for the traditional counterpart. However, the primary focus of the general categories often tends to show observable differences. An example of which is suitability of a specific material for the purpose of selection and use in a specific application. Identifying the need for an adequate amount of information coupled with a thorough analysis of the "emerging" materials does necessitate the need for a fundamental rethinking of why information of various kinds is essential and to whom and for what purpose is the specific information needed, essential and required. The information that is found,

established and subsequently collected, categorized and documented should not only be concise but also capable of being updated periodically. It should essentially represent areas of "valued" interest and much desired concern to representatives from both industry and policy makers. Over the years, the gradual development and emergence of new, improved and novel materials did get the much-needed interest, attention, participation and contribution from several researchers. This is evident from the fact that during the last four decades [*i.e.*, 1990 to present], several hundreds of papers have been published in the open literature on aspects specific to the development and emergence of new and improved materials made possible through a healthy synergism of novel changes in material chemistry coupled with an appropriate combination of innovative processing sequences to get the desired material.

The chapters contained in this bound volume attempt to provide an insight into the advances while concurrently addressing the potential areas of observable growth and resultant application of the new and improved materials resulting from a healthy combination of novel compositions and innovative processing techniques that were successfully developed and used for the synthesis of new and improved materials, referred to henceforth through this bound volume as "emerging materials". The manuscripts, or chapters, chosen for inclusion in this bound volume have been written by authors having varying backgrounds and experience in the domains specific to the synthesis, processing, manufacturing, experimentation, analysis, quantification and even modeling of materials and structures. This has essentially formed the basis of their writing style and technical content of their manuscript chosen for inclusion in this bound volume.

Overall, this bound book contains three sections. Each section, *i.e.*, **Section 'A', Section 'B'** and **Section 'C'** contains a few well laid-out technical chapters. In an attempt to make every effort to meet with the needs and requirements of the different readers, each chapter has been written and presented by one or more authors to ensure that it offers a clean, clear, cohesively complete and convincingly compelling presentation and discussion of the intricacies specific to their analysis, observations and resultant interpretations of their research and findings in a convincing manner.

In the **first section** of the book (**Section 'A'**), the focus is on "**METALS AND ALLOYS**" specific to the family of emerging materials. This section has five chapters. The first chapter [**Chapter 1**] introduces the interested reader to aspects pertinent to recent advances in friction stir welding of magnesium alloys for the purpose of selection and use in performance specific applications. The second chapter, [*i.e.*, **Chapter 2**], provides an in-depth analysis, in a cohesively complete and convincing manner, of the suitability of nickel-base shape memory alloys for selection and use in sensing applications. The follow-on chapter, [*i.e.*, **Chapter 3**] is devoted to presentation and healthy discussion of the intricacies specific to thermal and thermo-mechanical cycling studies on nickel-base shape memory alloys for selection and use in applications in both engineering and medical field. The authors present and adequately discuss all of the relevant and required aspects that are key for the purpose of selection and use of the nickel-base shape memory alloys in the two applications. The fourth chapter [**Chapter 4**] presents in a well laid out, neatly explained and convincing manner all of the related and relevant intricacies specific to the addition of nitrogen to Type 316L stainless steel with the prime objective of enhancing the performance of the chosen stainless steel at high temperatures when chosen for use in structural applications specific to fast reactors. All of the details and specifics are neatly presented and adequately discussed at all of the relevant and appropriate locations through the entire length of this chapter. The follow-on chapter on pressurized heavy water reactors [*i.e.*, **Chapter 5**] is thorough, exhaustive and illuminating in detail in a cohesively complete and convincing manner all of the intricacies specific to the evolution of zirconium alloy for use as pressure tubes in pressurized heavy water reactors. All

of the findings, observations and interpretations are neatly explained with the aid of appropriate micrographs to include both scanning electron micrographs and transmission electron micrographs. This is certainly a complete, well written and laid-out chapter that offers a wealth of information that is neatly explained using principles of Materials Science and Materials Engineering thereby significantly strengthening technical content of the chapter.

The **second section** of this book [*i.e.*, **Section 'B'**] is focused on "**COMPOSITE MATERIALS**" and includes six desirable and well laid out chapters. The first chapter in this section (*i.e.*, **Chapter 6**) attempts to provide the 'interested' reader with an overview of the desirable highlights specific to the selection and use of biomaterials and implants in orthopedics. Also provided and adequately discussed are key issues, or specifics, relevant to an evaluation of their future. This chapter can safely be categorized to be a healthy refresher to both the knowledgeable reader and 'learned' engineer while concurrently providing the novice and inquisitive learner useful information specific to the potential use of biomaterials and implants. In the following Chapter [*i.e.*, **Chapter 7**], key aspects specific to additive manufacturing of composite materials for use in biomedical applications are well presented and adequately discussed from both a scientific perspective and engineering viewpoint. In the next chapter [*i.e.*, **Chapter 8**], the theme for presentation and discussion is the key aspects specific to aluminum metal-matrix composites and magnesium metal-matrix composites. The contributing authors devote their attention and focus to providing adequate insight into developing an understanding of the role, importance and contribution of processing influences on corrosion properties of the chosen metal-matrix composites for the purpose of selection and use in environment-sensitive applications. In the following chapter [*i.e.*, **Chapter 9**], the contributing authors present their views, following a comprehensive study of aluminum nanocomposites that were developed by additive manufacturing for the purpose of selection and eventual use in both emerging and demanding automotive applications. In this chapter, the contributing authors also provide an adequate discussion of all intricacies specific to understanding processing influences on microstructural development, and microstructural influences in governing mechanical properties and resultant mechanical performance. In the following chapter [*i.e.*, **Chapter 10**], the contributing authors clearly present and thoroughly discuss lucidly all the key aspects and intricacies specific to enhancing the strength of aluminum-boron carbide composites to an adequately high level by the addition of magnesium. This enabled making the resultant composite material to be suitable for selection and use in a spectrum of applications in the automobile industry. In the following chapter [*i.e.*, **Chapter 11**], an adequate review of processing and fabrication of the sisal fibers-reinforced composite materials is neatly presented and adequately discussed with specific reference to understanding all of the intricacies specific to processing influences on microstructural development and the resultant influence of microstructure in governing mechanical properties and overall mechanical performance. This chapter based on both content and description can be considered to be educative, enlightening, and informative from the standpoint of an analysis and rationalization of the findings. In the same chapter [*i.e.*, **Chapter 11**] all of the key aspects specific to mechanical performance that result from the development of the engineered composites are well presented and adequately discussed.

The **third section** of this book [**Section 'C'**] is devoted to aspects both related to and relevant to "**OTHER MATERIALS AND TECHNIQUES**". In the opening chapter of this section [*i.e.*, **Chapter 12**], the contributing authors elegantly present and discuss the numerous benefits that arise from the selection and use of biomaterials and implants in orthopedics. The authors present and adequately discuss the basic principles behind biomaterials and implants and the overall benefits of selecting them for use in orthopedics. In the following chapter [*i.e.*, **Chapter 13**], the contributing authors make a comprehensive and complete review of "Smart

Hydrogels" with adequate emphasis given to both theory and applications in the domain specific to biomedical sciences. The contributing authors attempt to focus their review on studying and rationalizing the influence of basic theory in governing the selection and use of "Smart Hydrogels" in bioscience-dominated applications. In the following chapter [*i.e.*, **Chapter 14**], the contributing authors provide a neat and convincing review with appropriate discussion on the development of engineered iron-oxide-based nanomaterials for magnetic hyper-thermia. In the following chapter [*i.e.*, **Chapter 15**], the contributing authors provide a lucid and well-written overview of all of the intricacies specific to emerging and sustainable materials technology with an emphasis on fire safety. They present and adequately discuss the many attributes of the available alloys and materials for the purpose of their selection and use both in existing and emerging fire-safety critical applications. They also list and discuss the key considerations for both the existing materials and the newly developed materials while concurrently providing an overview of the future of the existing materials from the standpoint of eventual commercialization. In the following chapter [*i.e.*, **Chapter 16**], the authors provide an adequate review specific to recent advances in the unconventional machining of smart alloys in order to ensure their selection and use in critical manufacturing sectors. The following chapter [*i.e.*, **Chapter 17**] is well presented and appropriately discusses all the relevant aspects specific to critical parameters that exert an influence on the high strain rate deformation of engineering materials. The contributing authors provide a review of the published results from tests conducted on different materials using the pressure bar apparatus.

Overall, this archival monograph devoted to addressing the family of emerging materials provides a background that should enable an interested reader to comprehend with ease the immediate past, the prevailing present and the possible future, or emerging trends, and approaches in the domain specific to the gradual development. Also, the emergence of these materials with an emphasis on innovation is highlighted in an attempt to ensure their applicability for use in a wide spectrum of applications to include both performance-critical and non-performance-critical. Thus, based entirely on the contents included in this bound volume it can very well serve as a single reference book or even as textbook for the following:

1. Students spanning seniors in the undergraduate program of study in the fields of: (i) Materials Science and Engineering, (ii) Mechanical Engineering, and (iii) Manufacturing Engineering/Manufacturing Technology.
2. Fresh graduate students pursuing graduate degrees in: (i) Materials Science and Engineering, (ii) Mechanical Engineering, and (iii) Manufacturing Engineering/ Manufacturing Technology.
3. Researchers working in research laboratories and industries striving to specialize and excel in aspects related to research on materials science and engineering and the resultant development to ensure the emergence of new and improved products.
4. Engineers striving and seeking novel and technically viable materials for the purpose of selection and use in both performance-critical and non-performance-critical applications.

We certainly anticipate this bound volume to be of much interest and value to scientists, engineers, technologists, and even entrepreneurs.

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CHAPTER 1

Recent Advances in Friction Stir Welding of Magnesium Alloys for Use in Performance-Specific Applications

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Abstract: Magnesium is the sixth most abundant material in the earth's crust that finds its applications in the fields of automobiles, aerospace, and biomedical. With noticeable advances in the domain enveloping engineering and technology, there does exist a growing need for new and improved materials to meet the demands put forth by the industries spanning the aerospace and automobile sectors. One of the important requirements for a material is light in weight. Magnesium is one such promising material, which is lighter than aluminum making it an ideal candidate for selection and use in both performance-critical and non-performance critical applications in the domains specific to automobile, aerospace and even biomedical. There are various processing routes for the manufacturing of magnesium alloys, and there exists a need for the joining of the magnesium alloys. The conventional joining processes possess defects, such as porosity, which are detrimental to achieving acceptable to good mechanical properties. Friction Stir welding is one method of solid-state joining, which offers good properties of the weld. The technique of friction stir welding (FSW) operates by rotating and plunging a non-consumable tool into the interface of two workpieces that require to be joined. Promising advantages that are offered by friction stir welding (FSW) are eco-friendly, versatile, and energy efficient. This manuscript highlights (i) the friction stir welding processing technique, as well as recent and observable advances, (ii) the classification of the magnesium alloys, (iii) the welding tool and its influence on welding, microstructural development and mechanical properties of the friction stir welded magnesium alloy, (iv) welding parameters and its influence on governing the relationships between the weld and the workpiece, and (v) typical practical applications and the variants of friction stir welding (FSW).

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Keywords: Alloys of magnesium, Base metal (BM), Dwelling, Fixture, Friction stir welding, Magnesium alloy, Magnesium composite, Stir zone (SZ), Tool, Tool probe, Thermomechanical affected zone (TMAZ), Welding.

INTRODUCTION

With sustained and noticeable advances in technology, the world is gradually moving forward towards the selection and use of materials that are light in weight, have an excellent combination of mechanical properties and tribological qualities to offer coupled with other desirable characteristics. Magnesium is one such material, having acceptable mechanical properties and tribological qualities, coupled with chemical and biological capabilities and is low in weight [1]. Magnesium is often chosen for use in a variety of fields to include the following: (i) electrical industry, (ii) the aerospace industry, (iii) the vehicle industry, (iv) biomedical applications, and (v) industry that caters to the domain of sports, *i.e.*, sporting goods [2 - 4].

Magnesium makes up around 2.7% of the earth's crust and stands as the sixth most abundant element in the earth's crust [5]. The density of magnesium is 1.74 g/cm^3, which is two-thirds that of aluminum and one-fourth that of steel [6]. The properties of magnesium are summarized in Table **1.** Magnesium-based materials are extensively sought by firms for use in weight-critical applications essentially because of their low density coupled with high specific mechanical properties.

Table 1. Properties of magnesium [Reference 7].

Property	Magnesium
Density (g/cm^3)	1.74
Melting Point (K)	651
Specific Heat (J/kg. K)	1022
Young Modulus (10^{10} Pa)	4.43
Thermal conductivity (W/m. K)	167
Heat capacity (J/m^3. K)	1778
Thermal expansion rate (10^{-6}/K)	26.1

There are a variety of solid-state processing and liquid-state processing techniques, such as Additive Manufacturing (AM), Stir Casting, Melt Infiltration method, Spray forming, Friction Stir Processing, and Powder Metallurgy for the purpose of manufacturing magnesium alloys and magnesium composites. For the joining of a magnesium alloy and a magnesium alloy-based composite material with both comparable materials and different materials, friction stir welding,

resistance spot welding, laser welding, and diffusion bonding are all viable options [8 - 17].

Thomas and co-workers were the ones who initially created friction welding in 1991 [18]. When welding magnesium alloys and magnesium-based composites, Friction Stir Welding (FSW) offers a number of benefits that are not easily available with the other welding techniques. These benefits essentially include the following [19 - 22]:

(a) A fine microstructure,

(b) An absence of microscopic cracking,

(c) No loss of alloying elements during processing,

(d) Good dimensional stability and repeatability,

(d) Shielding gas is not required,

(e) High weld strength and toughness, and

(f) Capability of the weld to resist fatigue stress.

The basic idea behind friction stir welding is the same as that behind friction welding. During this process, heat is produced at the contact surface by the application of friction. The heat initiates the diffusion process at the surface where the two materials are to be joined. The application of a high-pressure force to these mating surfaces expedites the metal diffusion process and forms a metal- to-metal junction. This is the fundamental concept of friction welding [23 - 26].

Stages in Friction Stir Welding

The friction stir welding (FSW) process is broken up into three stages as shown in Fig. (**1**). The first stage is known as the Plunging phase. The second stage is known as the Dwelling phase, and the Third stage is known as the Welding phase [27].

The friction stir welding (FSW) technique essentially consists of only three stages, namely: (i) plunging phase, (ii) dwelling phase, and (iii) welding phase. Despite its seeming complexity, it is overall a very simple technique. A non-consumable revolving tool is used in the plunging process. The non-consumable revolving tool is composed of material that is stronger than the workpiece and has a shoulder that is bigger in diameter as well as a pin and will plunge into the workpiece to a depth that has been pre-programmed. This causes the generation of

Suitability of Nickel-base Shape Memory Alloys for Selection and use in Sensing Applications

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Abstract: In the prevailing era, an influential shape memory alloy (SMA) nitinol has emerged as a potentially viable and economically affordable material that is capable of playing a significant role in both existing and emerging technological applications spanning the domains of aircraft and aerospace, biomaterials in bioengineering, sensors in health monitoring, advanced manufacturing, and microelectromechanical systems (MEMS), to name a few. A high strain recovering capability coupled with super-elasticity are two key and essential characteristics of a "smart" material that distinguish it easily from its conventional counterparts. The phase transformation behavior shown by nitinol (NiTi) was found to be governed by intrinsic variations in temperature. In order to obtain the desired application-based functionality of this high performing material, potentially viable approaches include the following: (i) an alteration of its chemical composition, (ii) the addition of ternary elements and quaternary elements, and (iii) the use different processing treatments. These approaches are being constantly studied, carefully and systematically examined and frequently reported in the published literature. In this manuscript, an effort is made to present and discuss several of the recent advances specific to the NiTi-based shape memory alloy applications and its phase transformation behaviour when subject to processing treatments. The influence of compositional variation of the NiTi-based shape memory alloys (SMAs) and even its ternary variants and quaternary variants, coupled with the role and/or influence of different processing treatments on both macroscopic properties and microscopic properties is the focus. The emphasis on increasing the suitability of shape memory alloys (SMSs) for selection and use in a spectrum of sensing-related or sensing specific applications is highlighted and briefly discussed.

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Keywords: Compositional variation, NiTi-based shape memory alloy (SMA), Phase transformation behaviour, Processing treatments, Shape memory alloy (SMA).

INTRODUCTION

Shape memory alloys (SMAs) are in the prevailing and/or ongoing time period emerging as materials having a remarkable capability to recover higher strains while concurrently remembering their shape. These smart materials exhibit pseudo-elasticity properties, namely a combination of shape memory effect (SME) and super-elasticity, which are governed to an extent by their martensite to austenite phase transformation or austenite to martensite phase transformation.

Nickel-Titanium (Ni-Ti), referred to as nitinol, is a popular shape memory alloy (SMA) that has been researched during the last few decades, starting way back in the early 1950s. Though the shape memory effect (SME) was first discovered in Au-Cd alloy in 1951, Buehler and co-workers [1] in 1963, invented the shape memory effect (SME) in a Ti-Ni alloy. The titanium-nickel (Ti-Ni) alloy was having an equiatomic composition of titanium and nickel. Equiatomic or near-equiatomic NiTi shape memory alloy (SMA) can exist either in the austenite phase, martensite phase or the R-phase and concurrently reveal a dissimilar microstructure in these phases [2]. The R-phase normally forms as an intermediate phase for the austenite to martensite phase transformation and for the martensite to austenite phase transformation. Both of these transformations essentially involve two-stages for a specific composition. The R-phase is similar to the martensite phase, but does not provide the shape memory effect, or super-elasticity. At temperatures lower than the room temperature, *i.e.*, martensite start (Ms) temperature, a softer and deformable "twinned martensite" phase exists. This phase has a Young's modulus (E) and yield strength (σ_{YS}) that is lower than that of the austenite phase [2]. When the temperature of NiTi in the austenite form decreases gradually below the martensite start temperature (Ms), then the austenite NiTi starts to transform to martensite NiTi and the phase transformation is completed well below the martensite finish (M_f) temperature. The deformable "twinned martensite", when subject to pressure gets easily distorted to deformed "detwinned martensite". Upon heating the NiTi having martensite beyond the austenite start temperature (A_s), the NiTi begins to gradually transform to austenite and beyond the austenite finish (A_f) temperature, the entire austenite phase is formed. During phase transformation, the proportion of these phases tends to vary and the resultant mechanical properties are governed by these changes. Super-elasticity of a NiTi alloy is able to recover up to 8% of the total strain [3]. At ambient temperature above the austenite finish (A_f) temperature, super-elasticity favours the occurrence of martensitic phase transformation in the

NiTi alloy when subjected to a stress above a critical level. Upon relieving the stress, it tends to regain both its original shape and size spontaneously. When the NiTi alloy is deformed at temperatures below the martensite finish (M_f) temperature, heating it gradually above the austenite finish (A_f) temperature often results in regaining its undeformed shape and size. When a NiTi alloy is repeatedly subject to strain recovery cycles, it often results in actuation fatigue that generates irrecoverable deformation [4]. Suitable heat treatments have been found to increase the cycles-to-failure (N_f) of the chosen NiTi alloy when the temperatures are typically above 375°C.

The NiTi shape memory alloy (SMA) is found suitable for selection and use in a wide range of applications. A few of these applications include the following: (i) Sensors in health monitoring systems, (ii) Actuators in automobiles and aerospace, and (iii) Biomedical and biomaterials. The NiTi sensors are popular on account of their high sensitivity coupled with a wide range of strain measurements. Other noteworthy advantages of NiTi sensors are a low installation cost and overall compactness. The nickel-titanium (NiTi) and its combinatory shape memory alloy were produced by the addition of elements to result in the following: (i) Ni-Ti-Fe, (ii) Ti-Ni-Zr, (iii) Cu-Al-Ni, (iv) Ti-V-Al, (v) Ti-Ni-Cu, (vi) Ni-Ti-W, (vii) Cu-Al-Ni-Ti-B, (viii) Ti-V-Al-Y, (ix) Ti-V-Al-B, (x) Ti-V-Al Co, (xi) Ti-V-Al-Cu, (xii) Cu-Al-Ni-Hf, (xiii) Cu-Al-Ni-Mn, (xiv) Cu-Al-Ni-Gd, which have been tested and examined experimentally by researchers with the prime intent of establishing their suitability for selection and use in both sensor and actuator applications [1, 2, 4 - 24]. An alteration in the chemical composition, addition of ternary elements or quaternary elements and processing, to include both heat treatment and surface treatment, have been successfully tried and reported to offer improved macroscopic properties and microscopic properties of the material [2, 4 - 17, 19, 20, 22 - 24]. Considering their unique super-elastic behavior, the NiTi shape memory alloys have been chosen for use as self-expanding vascular stents and orthopaedic bone staples. Due to concerns about allergy to nickel, nickel-free β-type titanium-base shape memory alloys (SMAs) were developed. However, their recoverable strain is less than 5 percent, approximately half that of the NiTi alloys.

This manuscript broadly focuses on a satisfactory introduction to nickel-titanium (NiTi)-based shape memory alloys (SMAs), potential viable approaches to enhance both their macroscopic properties and microscopic properties while concurrently making them suitable for selection and use in a spectrum of sensing-specific or sensing-related applications. The effects of different processing treatments on this "new era" material are also highlighted. The following section provides a brief overview of the background specific to shape memory alloys and the inspiration leading to its invention.

Thermal and Thermomechanical Cycling Studies of Nickel-Based Shape Memory Alloys for Engineering and Medical Applications

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Abstract: Shape memory alloys (SMAs) are those that can return to their initial shape after deformation under a stimulus, such as temperature or stress. They are capable of recovering deformations of up to 8%. Generally, the martensitic transformation is reversible in nature and the shape memory alloys exhibit two unique characteristics, super-elasticity effect (SE) and shape memory effect (SME), depending on whether these properties/responses are brought on by stress and temperature, respectively. Since the shape memory alloys undergo full cycling, they transform from austenite to martensite at temperatures between martensite finish and austenite finish. However, partial cycling refers to heating above the austenite start temperature but below the austenite finish temperature followed by cooling to below the martensite finish temperature. The phase transformation is partial before it is complete, consequently, only smaller amounts of the phases undergo a phase transition. Based on the operating temperature window and the transformation temperatures of the alloy, partial cycling can be divided into three categories. This chapter discusses the various types of cycling, *i.e.*, thermomechanical, thermal, and partial cycling behavior of nickel-titanium-based shape memory alloys.

Keywords: Austenite and martensite, Partial transformation, Shape memory alloys, Transformation temperatures.

INTRODUCTION

Shape memory effect was first discovered in 1931 by A. Olandar in a gold-cadmium alloy and reported the phenomenon that was observed as a rubber-like behaviour [1]. After almost a decade, the metastable β-based copper-zinc shape memory alloy was discovered by A.B. Greninger and V.G.Mooradian [2], and the

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concept of thermoelastic martensite was then proposed by Kurdjumov in 1949 to explain the reversible nature of transformation in copper-aluminium and copper-zinc alloys [3]. The term shape memory effect was coined by Chang and Read in 1951 to describe the thermoelastic behaviour of shape memory alloys [4]. Nevertheless, none of them made any inroads into the commercial market until 1963 when Buehler W.J. and his co-workers from the Naval Ordnance Laboratory in the U.S. discovered the nickel-titanium (NiTi) shape memory alloy, which is now commercially known as Nitinol [5].

Currently, shape memory alloys are widely used in many industries, including automobile, aerospace, aviation, robotics, biomedical, textile and garment, defence, and electrical and electronics. Recently, the National Aeronautics and Space Administration (NASA) has developed a smart wheel made of a Ni-rich nickel-titanium shape memory alloy for interplanetary and lunar exploration purposes [6]. It is forecast that by 2023, the global market for shape memory alloys will surpass USD 20 billion [7]. Till date, many other new shape memory alloy systems exhibiting shape memory characteristics have been discovered, developed, and used. A few of these are listed in Table **1**.

Table 1. Popular alloy systems exhibiting shape memory characteristics (Mohd Jani *et al.*, 2014; Wayman, 1993).

Alloy System	Alloy Composition
Nickel-titanium	NiTi, NiTiCu, NiTiNb, NiTiFe, NiTiCo, NiTiPt, NiTiPd, NiTiHf, NiTiZr
Copper	CuZn, CuSn, CuZnAl, CuAlNi, CuAlBe
Iron	FePt, FePd, FeMnSi, FeNiC
Titanium	TiTa, TiNb, TiMo, TiTaAl, TiTaSn, TiTaSi
Others	ZrCu, ZrCuNiCo, AuCd, AgCd, CoNiAl, CoNiGa, NiMnGa, InTl, NiAl, InCd, RuTa, RuNb, AuTi, MnCu,

The following are the characteristics that are significant in the context of shape memory alloys;

a) Phase transformation temperatures (austenite start, austenite finish, martensite start, martensite finish);

b) Recovery strain;

c) Recovery stress; and

d) Hysteresis

Phase Transformation Temperatures

When the shape memory alloys are cooled down from the austenitic region, they undergo the thermoelastic martensite transformation over a range of temperatures. The temperatures at which the martensitic transformation start and finish are referred to as martensite start and martensite finish temperatures, respectively. Similarly, austenite start and austenite finish temperatures refer to the temperatures at which the austenite transformation begins and ends, respectively, during heating. The intermediate phases, including R-phase, may also be present in the material depending on the alloy system and thermomechanical treatment. The relevant transformation temperatures for R-phase formation are referred to as R_s and R_f, respectively.

There are many factors that affect the transformation temperatures of shape memory alloys, for example, the alloy composition, microstructure, degree of order, thermal and thermomechanical treatments, as well as defect density (dislocations and point defects) [8]. The traditional techniques to determine the transformation temperatures of shape memory alloys include differential scanning calorimetry (DSC) and electrical resistivity (ER). Other techniques, including dynamic mechanical analyser (DMA) [9] and dilatometry [10], are also employed to determine the transformation temperatures.

Recovery Strain

Upon heating above the austenite start temperature, it is possible to recover some of the strain imparted during the deformation of a shape memory alloy in the martensitic state (up to 8%). Various factors influence the magnitude of recovery strain, for instance, alloy chemistry, thermal and mechanical processing, stress, and working temperature. A range of testing methods can be used to measure the recovery strain, such as dynamic mechanical analysis, bend test, and thermomechanical cycling test. The thermomechanical cycling test (thermal cycling under constant stress) is the most common method for determining recovery strain. The magnitude of the recovery strain can be determined by comparing the maximum and minimum strains found on the strain versus temperature plot.

Recovery Stress

The recovery stress is generated by elastic recovery during deformation. The shape memory alloys on deformation generate stresses, which are stored in the martensitic variants during the deformation, on heating above the austenite start temperature under a constraint. It can be used to provide support or external confinement to both mechanical and civil structures.

Nitrogen Additions to Type 316L Stainless Steel to Enhance its High Temperature Performance for Structural Applications in Fast Reactors

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Abstract: The high-temperature performance of the nitrogen added 316L stainless steels including the high-temperature mechanical properties, workability and weldability have been investigated in detail by systematic studies with nitrogen contents in the range of 0.07-0.22wt.%. Strength and creep resistance were found to increase with increasing nitrogen content at room and elevated temperatures. However, resistance to creep-fatigue damage, fracture toughness, resistance to fatigue crack growth, workability, and weldability were found to degrade beyond 0.14wt% of nitrogen content at service temperature. Therefore, the maximum nitrogen content in 316L stainless steel is recommended to be 0.14wt% for better high-temperature performance in structural applications of fast reactors.

Keywords: Creep, Creep-fatigue interaction, Dynamic materials model, Fracture toughness, Fatigue crack growth, Fractographs, Hot cracking, Nitrogen alloying, Tensile properties, Workability, Weldability, 316LN Stainless Steel.

INTRODUCTION

Type 316 Stainless Steel and its variants are the major structural materials chosen for fabricating structural components of fast reactors. Low carbon variety is chosen mainly to avoid sensitization during welding and later failure due to stress corrosion cracking in corrosive environments. The high-temperature performance of the 316L stainless steel can be significantly improved by adding nitrogen beyond 0.06 wt%. Nitrogen-added 316L stainless steel designated as 316LN stainless steel having the best combination of strength, toughness and corrosion resistance already found applications in areas as diverse as cryogenic, nuclear, power generation and chemical industries. The beneficial effect of adding nitrogen to 316L SS arises due to the higher solubility of nitrogen in the austenitic

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matrix, reduction in stacking fault energy causing larger separation in partial dislocations, strong solid solution strengthening caused by strong elastic interaction between interstitial nitrogen and the dislocations and retardation of coarsening of $M_{23}C_6$ type carbides [1]. It is well established that the addition of nitrogen to austenitic stainless steel has a beneficial effect on corrosion resistance.

The increase in nitrogen content decreases the defect density in the space charge layer, thereby promoting the enrichment of chromium within the passive film accompanied by the formation of a more stable film with fewer defects [2]. Nitrogen addition up to 0.16 wt% increases resistance to sensitization by retarding the coarsening rates of carbides because it reduces the diffusivity of chromium and carbon. However, the upper limit of nitrogen content needs to be determined for the structural applications in fast reactors considering the effect of nitrogen content on the high-temperature mechanical properties. The ease of fabricating nitrogen alloyed 316L SS into useful structural components and products for fast reactor applications by conventional metal forming processes and welding also needs to be considered for determining the optimum nitrogen content.

Type 316LN SS containing 0.02-0.03 wt% carbon and 0.06-0.08 wt% nitrogen has been used for fabricating all major structural components of the 500 MWe Prototype Fast Breeder Reactor (PFBR) that is in the advanced stage of commissioning at Kalpakkam [3]. The design life of the PFBR is 40 years. As the design life of future sodium-cooled reactors is envisaged to be enhanced to 60 years, structural material with improved high-temperature mechanical properties is required. To meet the enhanced life requirements of structural components, type 316 LN SS with nitrogen additions in the range of 0.07-0.22wt% have been developed. The varying heat of 316 LN SS with varying nitrogen content was produced at MIDHANI Limited, Hyderabad.

The effect of nitrogen content on the tensile, creep, low -cycle fatigue behavior, creep-fatigue interaction, fracture toughness, fatigue crack growth behavior, workability, and weldability was investigated in detail. The workability of steels has been studied by generating processing maps using Dynamic Material Models (DMM). The safe processing domains have been identified for producing 316LN stainless steel in various forms using industrial metal forming processes. During the welding of austenitic stainless steel, it is well known that the formation of ferrite as the primary phase during solidification reduces hot cracking susceptibility. Nitrogen addition to the above steel only decreases the occurrence of primary ferrite solidification and hence promotes hot cracking susceptibility. Further, nitrogen addition enhances the segregation of phosphides and sulphides at the grain boundaries which also promotes hot cracking. Therefore, weldability studies involved investigating the hot cracking behavior. The present chapter

discusses the influence of nitrogen on high-temperature mechanical properties, workability and weldability of 316LN SS.

MATERIAL DETAILS

Four commercial scale heats of 316LN SS, containing 0.07, 0.11, 0.14 and 0.22wt.% nitrogen (henceforth designated as N07, N11, N14 and N22 respectively) were produced by double melting technique. The primary melting was carried out in air induction melting (AIM) followed by secondary remelting using electro slag refining (ESR) processes. Nitrogen content was varied with the addition of nitrided ferrochrome.

The amount of all the other elements was kept unchanged in all heats. The carbon content in all heats was maintained at around 0.03 wt.%. The ESR ingots were hot forged to slabs which were then hot rolled into plates of 22 mm thickness. The chemical composition of the four heats of 316LN SS is given in Table **1**.

Table 1. Chemical composition (in wt.%) of nitrogen alloyed 316LN austenitic stainless steels.

Designation	C	Cr	Ni	Mo	N	Mn	S	P
N07	0.03	17.5	12.2	2.49	0.07	1.7	0.0055	0.013
N11	0.03	17.6	12.2	2.51	0.11	1.78	0.0055	0.015
N14	0.03	17.5	12.1	2.53	0.14	1.74	0.0041	0.017
N22	0.03	17.5	12.3	2.54	0.22	1.7	0.0055	0.018

Rectangular blanks cut from the rolled plates in the rolling direction, were solution annealed at 1363 K for 30-60 minutes followed by water quenching. Equiaxed grains free of precipitates and delta-ferrite have been observed. The average grain size of four heats, measured through the mean intercept method is found to be in the range 89 ± 13 μm.

INFLUENCE OF NITROGEN CONTENT ON THE TENSILE BEHAVIOUR OF 316LN STAINLESS STEEL

The tensile behaviour of 316LN stainless steel has been investigated with nitrogen content varying from 0.07 to 0.22% [4]. Fig. (**1**) shows the combined effect of temperature and nitrogen content in the variations of yield strength and ultimate tensile strength (UTS) for the four variants of nitrogen alloyed 316LN stainless steel. In general, strength increased with increasing nitrogen content at any test temperature (Fig. **1a**). For the range of nitrogen contents examined in the present study, an increase in yield strength of 70 MPa and 40 MPa per 0.1 wt. % N increase has been observed at 300 K and 923 K respectively (Fig. **1b**). Three

On the Evolution of a Zirconium Alloy for Use as Pressure Tubes in Indian Pressurized Heavy Water Reactors

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Abstract: In the early generation of the Indian 220 MWe pressurized heavy water reactor (PHWR220), Zr-2.5Nb pressure tubes (PT) were manufactured from double-melted (DM) ingots. Later on, quadruple melted (QM) ingots were used to achieve enhanced performance. These pressure tubes were fabricated by hot extrusion followed by double pilgering with intermediate annealing and this fabrication route is designated as an old route (OR). These tubes have performed reasonably well. However, some of these tubes showed higher in-reactor deformation. Subsequently, both alloy chemistry and manufacturing practice were revisited and changes in alloy chemistry and ingot diameter, mode of hot working for breaking the cast structure and hot extrusion of billets with higher extrusion ratio and single pilgering steps have been employed. This route is designated as a new route (NR) and is being used for manufacturing pressure tubes for the current generation of 220MWe pressurized heavy water reactors.

Over the years, changes in Chlorine (Cl), Carbon (C), Phosphorous (P), Iron (Fe) and Hydrogen (H) specification and narrowing down the specification for Niobium (Nb) and Oxygen (O) have been implemented to exploit their beneficial effect on in-reactor deformation and hydrogen pickup. The changes in manufacturing practices had resulted in changes in microstructure and texture. In the old route (OR), pressure tube (PT) microstructure was characterized by the presence of discrete beta-phase precipitates along the interfaces of alpha lamellae while the new route (NR) pressure tube (PT) exhibits more continuous beta film and relatively coarser α lamellae. In terms of crystallographic texture too, the new route (NR) pressure tubes (PTs) had higher F_T values (in the order of 0.65) in comparison to old route (OR) pressure tubes (PTs) ($F_T \sim 0.55$ to 0.6).

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Because of crystallographic and microstructural anisotropy, the tensile behavior of this material is also anisotropic with the transverse direction exhibiting higher flow stress and lower ductility at and below reactor operating temperatures. The transverse tensile strength of pressure tube (PTs) fabricated from new route (NR) is higher than that fabricated from old route (OR). The fracture toughness of pressure tubes (PT) manufactured from quadruple melted (QM) ingots are significantly higher than that of the pressure tubes (PTs) manufactured from double melted (DM) ingots, which is attributed to the deleterious effect of Chlorine (Cl), Carbon (C), Phosphorous (P) and their complexes. The variation in fracture toughness of pressure tubes (PTs) was evaluated as a function of temperature, hydrogen content and hydride orientation. The hydrided material exhibited a typical S curve showing lower-shelf, transition and upper shelf regimes. Delayed hydride cracking velocity and threshold stress intensity factor were determined as a function of temperature, direction of approach to test temperature and hydride orientation. Threshold stress for hydride reorientation (σ_{th}) determined using *ex-situ* and *in-situ* methods between 250 and 300°C was observed to decrease with an increase in temperature. Thermal creep behavior was investigated for these tubes at 350, 400 and 450 °C at different stress levels and comparison of the minimum creep rate and the rupture life is presented. This article describes the evolution of the alloy chemistry, microstructural features, texture and mechanical properties and hydride induced embrittlement of the pressure tubes (PTs) used in Indian pressurized heavy water reactor (PHWR) and life extension approaches. An attempt has been made to rationalize the observed properties in terms of alloy chemistry and microstructure.

Keywords: Creep, Delayed hydride cracking, Fracture, Life extension, Microstructure, Pressure tube, Tensile, Zirconium alloy.

INTRODUCTION: NUCLEAR REACTOR

The standard of living of any country is directly related to the per capita energy consumption. Fossil fuel continues to be the main source of energy. However, due to limited resource of the fossil fuels and harmful effect of the gases released during its combustion to the environment, all sources of energy including nuclear are being tapped. Nuclear Power Plant facilitates the generation of power. A typical fission based nuclear power plant comprises a reactor, a steam generator, a turbine, a condenser and safety systems. The reactor contains fuel, sustains nuclear chain reaction to produce heat and holds the nuclear waste [1].

Based on the energy of neutrons used to cause fission, nuclear reactors are called fast and thermal reactors. Thermal reactors contain fuel, a moderator, control rods, a coolant and safety systems. The moderator is required to thermalize the high energy neutrons so as to improve the probability of fission reaction. The heat produced in a nuclear reactor is extracted by the coolant to produce steam, which is fed to one or more turbines that energize an electric generator to produce electricity that is fed to the electrical grid. The cooling towers reject excess heat from the turbine section to the cooler surrounding. Adequate safety systems are in

place to ensure safe operation. The efficiencies of nuclear power plants are around 33-37%, which is comparable to fossil fuel power plants. Modern designs like the Generation IV nuclear reactors operate at higher temperatures and could achieve above 45% efficiency [1]. Presently, about 11% of the world's total electricity is generated by Nuclear fission reactors [1] and has vast untapped potential to contribute more especially to replacing the ageing fossil fuel power plants and achieve emission targets.

Pressurized Heavy Water Reactor

The generation of steam is common for all nuclear reactors and is often used to classify various reactor systems. Water cooled reactors like pressurized water reactor (PWR), pressurized heavy water reactor (PHWR) and boiling water reactor (BWR) use light / heavy water as coolant and moderator [2]. The PWR and PHWR use two loops called primary and secondary loops. The primary loop containing liquid water under high pressure is used to extract heat from the reactor core and serves as a heat source for the heat exchanger. The water at a lower pressure circulating in the secondary loop extracts heat from the heat exchanger, and generates steam that is fed to the turbine. The BWR heat transforms the water in the core directly to steam. The technology for the manufacture of large pressure vessel required for PWR and BWR has been in use in few countries only. The PHWR circumvents the need for large pressure vessel and uses tubes as part of primary heat transport loop instead of vessel of PWR. The cost of building the PHWR is also lower than the reactor systems that require large pressure vessel.

The PHWR was developed in Canada in 1950s and is popularly known as the CANDU (Canada Deuterium Uranium) Reactor [1]. The use of natural uranium (0.7% U^{235}) oxide as fuel in PHWRs requires a more efficient moderator. The energy produced per kilogram of mined uranium in PHWR is higher compared to other designs, but it also produces a much larger amount of spent fuel per unit energy generated. In PHWR, the heavy water moderator is enriched instead of the fuel [1]. The pressure tubes in PHWR serve as miniature pressure vessels, and contain the fuel bundles and hot pressurized coolant. The moderator is separate from coolant and is contained in the calandria vessel. The pressure tube design allows refueling of the reactor without shutting down, by isolating individual pressure tubes from the cooling circuit.

Advanced PHWR design like the Advanced CANDU Reactor (ACR) is cooled by light water and uses slightly-enriched fuel [3]. CANDU reactors may be run on recycled uranium obtained from reprocessing of the PWR spent fuel, or a blend of this and depleted uranium. Thorium could also be used as fuel. Indian designed

State of the Art in Additive Manufacturing of Metal Matrix Composite for Use in Performance-Specific Application

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Abstract: Metal matrix composites (MMCs) have proved themselves a reliable alternative to different metals and their alloys due to their high strength-to-weight ratio, high durability; high wear corrosion resistance, high hardness and other mechanical properties. In the conventional approach, different methods like liquid-state processing (stir casting, squeeze casting *etc*.), solid-state processing (consolidation, physical vapour deposition or PVD, powder bending *etc*.), and *in-situ* processing are being used to manufacture metal matrix composites (MMCs). Injection molding and other *in-situ* processing are highly dependent upon particle size and morphology. Particle agglomeration is a common problem for liquid and solid-state processing. Again, these inhomogeneous second-phase particles influence crack initiation and propagation, thermal mismatches, residual stresses, and dislocation, making the subtracting or machining process challenging to perform. By observing these issues with the conventional approach, additive manufacturing can be considered an alternative technique to fabricating metal matrix composite. It is reported that 3D printing cannot only sort out the matrix/reinforcement bonding issues observed during conventional manufacturing processes but is also capable of providing a uniform distribution of reinforcement inside the metal matrix. Additive manufacturing allows the fabrication of functionally graded composites with any geometrical complexity, higher accuracy, and minimum production lead time. However, challenges like lack of fusion, rapid cooling, poor surface morphology and texture restrict the additive manufacturing processes to manufacturing a sound product. The current chapter summarises the recent development in manufacturing metal matrix composites (MMCs) using different additive manufacturing processes.

Keywords: Additive manufacturing (AM), Challenges, Conventional manufacturing process, Metal matrix composite (MMC), Mechanical properties, Microstructural properties, Reinforcement.

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INTRODUCTION

Additive manufacturing is an innovative and powerful technology that hugely impacts today's industrial manufacturing hub. Additive manufacturing, as the name implies, can be a consecutive layer-based production technology. There is mainly a 'Three Pillar Concept' by which a desirable product can be made.

I. Subtractive Manufacturing Pillar – Conventional and Non-Conventional Machining.

II. Constant Volume Pillar – Metal Forming, Casting.

III. Additive Pillar – Joining, 3D Printing.

Among these techniques, additive manufacturing (AM) or 3D printing is a very new emerging technique that blows our minds with customer satisfaction with the highest integrity. David E.H. Jones laid out the very first concept of the 3D printing technique in 1970. In the 1980s, its materials and equipment were developed, and then it was known as, 'Rapid Manufacturing' or 'Generative Manufacturing', but in future, it is realized that 3D printing somehow differs from rapid manufacturing. Rapid manufacturing and rapid prototyping are the two application levels of additive manufacturing. In the 1990s, several AM techniques were developed, and extensive research is still going on to optimize these techniques to manage mass production in the industry.

OVERVIEW OF ADDITIVE MANUFACTURING

General Introduction to Additive Manufacturing

A layer-based automated fabrication technology called additive manufacturing (AM) uses 3D CAD (computer-aided design) data to create scaled, three-dimensional physical objects without the aid of path-dependent tools. Unlike conventional machining, forming, and casting operations, which involve taking material from a stock item (subtractive manufacturing) or pouring it into a mould and shaping it with hammers, dies, and presses. Any complex shape can easily be made by additive manufacturing (AM). It provides a way to print a product through a simple and economical path. A 3D item or model is created by layering consecutive layers of powder, liquid, sheet material, polymer or other materials by additive manufacturing equipment, which reads data from the CAD file. One can say that additive manufacturing is a 2.5D process as its Z-axis has a very small thickness (small layer). In this decade, rapid prototyping is getting converted into rapid manufacturing and producing the parts in mass-customized products with the help of additive manufacturing. A 3D-printed part is called a product or the

final desired part if it shows all the characteristics and functions allocated to it during the product development process [1]. Additive manufacturing has attracted many researchers due to its vast application in the field of medical, automobile and aerospace industries.

Terminology

The term Additive Manufacturing (AM) gained its popularity in the 2000s. The term AM is surrounded by several technologies as well as subsets like:

I. Rapid: Rapid Manufacturing, Rapid Technology, Rapid Prototyping, Rapid Tooling.

II. Additive: Additive Manufacturing, Additive Layer Manufacturing, Additive Digital Manufacturing.

III. Layer: Layer Based Manufacturing, Layer Oriented Manufacturing, Layer Manufacturing.

IV. Digital: Digital Mock and Fabrication.

V. Direct: Direct Tooling, Direct Manufacturing,

VI. 3D: 3D Printing, 3D Modelling.

Development and Fundamentals of Additive Manufacturing

The most outstanding goal of virtually any development method is to minimize the time and energy of changing a thought into a reality. Since the 1970s, manufacturers have centred on 'TQÇS', where T stands for time to market, Q stands for quality, C stands for cost, and S stands for service. In parallel to those changes, engineers have developed rapid manufacturing, especially with the help of computers, the Internet and information management technologies. Advanced manufacturing technology has climbed to a better level in the last decade as various new philosophies like lean production (LP), virtual manufacturing (VM), agile manufacturing, and virtual enterprise (VE) came to the manufacturing industry field. Rapid prototyping (RP) or additive manufacturing (AM) is one of the latest technologies. In several aspects, additive manufacturing is an industrial sector instead of a technology employed in many different sectors. Up to date market strategy analysis says that there are four stages within which the life cycle of an industry is bounded: introduction, growth, maturity and decline. Tiny sales, low rates of market penetration, low market and high prices define the introduction stage. Additive manufacturing spent a significant amount of time in this stage dealing with expansion constraints, industry-wide innovation

Additive Manufacturing of Composite Materials for Use in Biomedical Applications

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Abstract: The life of human beings is moving at a breakneck pace, with a fast-moving life demanding the need for devices for use in biomedical applications, which attracts the interested researcher to work on ensuring novel breakthroughs. Processing of biomaterials is one of the key factors that will exert an influence on impacting the attributes of a biomaterial. Additive manufacturing is one of the promising routes by which layer-by-layer creation of parts takes place from a computer-aided design (CAD) file. Parts that cannot or are difficult to manufacture by other processing routes can be easily manufactured using the technique of additive manufacturing (AM). Parts, such as (i) stents, (ii) customized prosthetics, (iii) organs, and (iv) implants can be easily manufactured using the technique of additive manufacturing (AM). With noticeable advances in the domain specific to additive manufacturing, the biomedical field is being revolutionized, and viable solutions to difficult problems are being put forth with ease, and the resultant by-products offer a combination of acceptable to good properties. The key benefits of the technique of additive manufacturing (AM) are low cost, minimal material waste, and enhanced product reliability. This study explores recent developments in both alloys and composite materials processed by the techniques of additive manufacturing for selection and use in biomedical applications. This review provides a highlight of the different additive manufacturing techniques with specific reference to biomedical applications and additive manufacturing of titanium alloys, the Co-Cr alloy, the magnesium alloys and their composite counterparts. Multidisciplinary research will be required to meet and overcome any and all obstacles while concurrently fulfilling the potential of additive manufacturing (AM) in the years ahead.

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Keywords: Additive manufacturing, Biomaterials, Binder jetting, Biomaterials, mechanical properties, Co-Cr alloy, Composite material, Direct energy deposition (DED), LENS, Material extrusion (ME), Material jetting, Powder bed fusion (PBF), Sheet lamination, SLA, Vat polymerization, 3D printing.

INTRODUCTION

With noticeable advances in medical sciences, new techniques are being gradually developed for the treatment of various issues specific to human beings. With the development and emergence of new techniques, there does exist a need for advanced manufacturing techniques that will adequately compensate for the need for medical sciences for devices, parts and/or components. For a large majority of the prevailing medical devices, such as (i) prostheses, (ii) surgical instruments, and (iii) general equipment/tools, used by medical professionals, the conventional manufacturing processes of CNC (Computer Numerical. Control) machining and injection molding have been used. This is essentially a subtractive manufacturing technique in which the design of a medical device is cut out from a piece of manufactured block [1]. Additive manufacturing (AM), or 3D Printing, is an advanced technique in which a 3D part or component is made by sequential deposition layer upon layer.

The development of 3D printing for biomedical applications is shown in Fig. (**1**). The 3-D printing was first introduced in 1984 by Charles Hull, which resulted in the technique of stereolithography (SLA) being invented [2]. In 1988, Robert J. Klebe first demonstrated 3D printing, which was done by using cyto-scribing technology on a Hewlett-Packard (HP) inkjet printer [3]. In 1999, the first 3D printing bladder containing cells was made possible by utilizing layers by Odde and Renn [4].
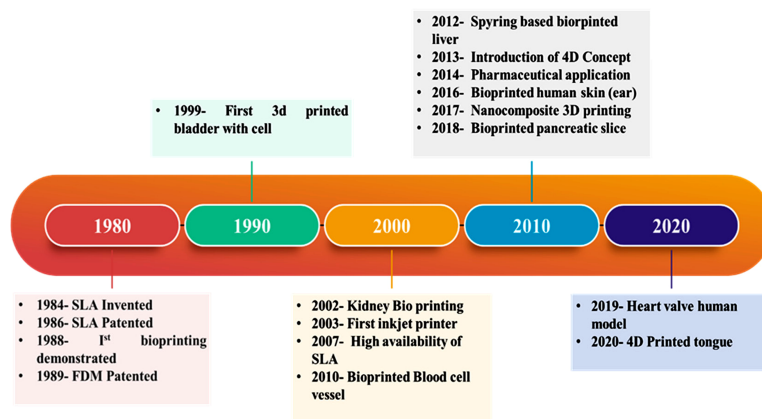
Fig. (1). History of development of 3D printing for biomedical applications.

In 2002, the bio-printing of a kidney was made possible using an extrusion-based technique [5]. Wilson and co-workers [6] remodified a standard Hewlett Packard (HP) printer and developed the first inkjet printer in 2003. There was availability of stereolithography (SLA) in 2007. The bio-printed blood vessels were made in 2010 [7]. The years 2012-2018 saw the development and emergence of the 4D printing technique, and the printing of some critical parts was made possible [8, 9]. Noor and co-workers [10] used 3D printing to develop heart and cardiac patches using a bio-ink material. Additive manufacturing (AM) did enable in manufacturing of parts that had an intrinsic shape, where the need for precision was both essential and required.

The basic steps involved in additive manufacturing (AM) are as follows: (i) Data acquisition, (ii) Image Processing, (iii) Printing of biomedical device/part, (iv) Sterilization and post processing, and (v) Implantation. This is shown in Fig. (**2**)**.**
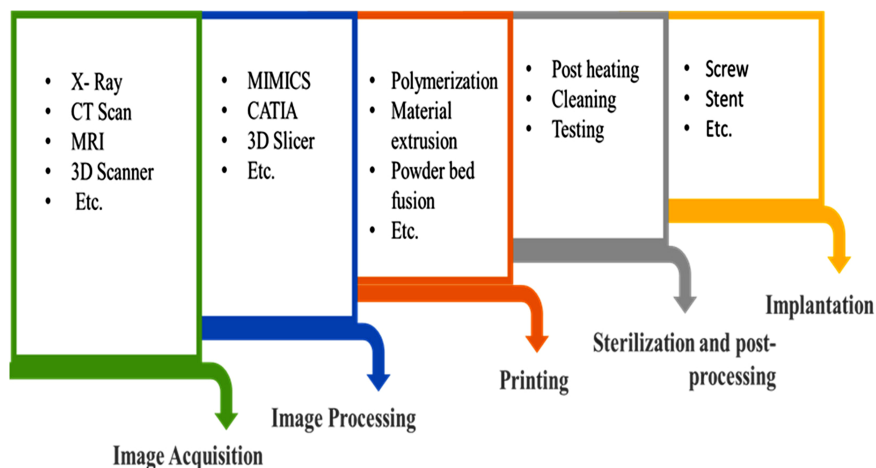
Fig. (2). Steps involved in additive manufacturing (AM) of a biomedical device/part.

Image Acquisition is the first step in Additive Manufacturing (AM) in which the image is retrieved from the source. The software used in image acquisition are X-Rays, CT scan, MRI, and a 3-D scanner [11]. Image processing is the next step in which image segmentation takes place, *i.e.*, converting the physical image into a digital image. The software often chosen for use in image segmentation are (i) MIMICS, (ii) CATIA, and (iii) 3-D slice. A DICOM format file is obtained using this step, which is further converted into an STL format. The next step is printing of the part in which a suitable method is chosen depending on the following: (i) the material, (ii) the requirements of the part, and (iii) printing parameters. Materials, such as hydrogels, biomaterial, cells and bio-ink, can be chosen for use

Aluminum Metal Matrix and Magnesium Metal Matrix Composites: An Insight into Processing Influences on Corrosion Properties for use in Environment- Sensitive Applications

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Abstract: The limitations of engineering are mainly attributed to the limitations in the availability of materials that can deliver all the desired properties for a given end application. But, due to the development of composites, there has been an enormous advancement in industries like automobile, sports, power generation, defense, and aerospace. Metal Matrix Composites became one of the sought-after materials because of their high specific properties, such as strength-to-weight ratio. Even though these materials exhibit enhanced mechanical and thermal properties, their usage is still limited. Some of the reasons include the scaling up of processing and the uncertainty in their joining and corrosion behavior. This study attempts to bring together information on metal matrix composites processing and corrosion behavior, mainly focusing on Magnesium and Aluminum metal matrix composites.

Keywords: Aluminum composites, Corrosion, Magnesium composites, Mechanical properties, Metal matrix composites, Processing methods.

INTRODUCTION

Industries like aerospace, automobiles, transportation and electronics have been using metals as their primary manufacturing materials for a long time. Even though metals offer a desirable combination of mechanical properties such as tensile strength elastic modulus, ductility, malleability, toughness, and fatigue resistance, the quest for materials that also provide additional desired properties like corrosion and creep resistance, tailorable coefficient of thermal expansion (CTE), *etc*. steadily progressed. Some noticeable outcomes of the works of resea-

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rchers were the compositional tailoring of metals leading to multicomponent alloys and the emergence of metal matrix composites.

The progress of modern engineering has been significantly improved by the development of alloys. Materials like magnesium alloys, well known for being the lightest structural alloys, and iron alloys like stainless steel that demonstrate exceptional magnetic, thermal, and mechanical properties and corrosion resistance provided engineers to develop products with significant functionalities. Yet, magnesium alloys still exhibit low strength and elastic modulus comparatively. Ceramic materials exhibit high strength and toughness but are not readily used in manufacturing because of their low ductility, poor toughness, *etc*. When metals or metal alloys are reinforced with ceramic materials like silicon carbide (SiC), aluminum (III) oxide (Al_2O_3), aluminum nitride (AlN), yttrium(III) oxide (Y_2O_3), and titanium diboride (TiB_2), resultant materials inherit properties from their constituent materials leading to highly desired combination of properties. Often, the improved hardness and yield strength of metal matrix composites reduce the materials' ductility and fracture toughness especially when the reinforcement is in micron and sub-micron length scale. Lately, metals like titanium (Ti), copper (Cu), *etc*., and carbon fibers are being used as alternate reinforcement materials. Most properties of composites are improved over their constituent materials, but sometimes, these reinforcements act as inert conductors of electricity, allowing the possibility of galvanic corrosion in metal matrix composites. This decreases the overall corrosion resistance of metal matrix composites.

The rule of mixtures is generally used to estimate the mechanical properties of composites. However, corrosion properties can be entirely different from either of their constituent materials, depending on the different forms of corrosion (*e.g.*, uniform corrosion, pitting corrosion, galvanic corrosion, crevice corrosion, stress corrosion cracking, microbial corrosion *etc* [1]) observed in metal matrix composites. Hydrogen evolution test, a widely used electrochemical test to determine the corrosion rate of magnesium-based materials, optical microscopy and scanning electron microscopy are suggested to estimate the corrosion properties of composites. Due to the difference in electrochemical potential between the reinforcements and matrix materials, galvanic corrosion is one of the most significant corrosion mechanisms observed in metal matrix composites, along with the chemical degradation of interphases and reinforcements-induced corrosion. In this review, we will start with what composites are and their classification as a brief introduction. Then the concept of galvanic corrosion in metal matrix composites will be introduced, followed by an introduction to polarization curves, which are significant in estimating the corrosion properties of metal matrix composites. Finally, the effects of processing methods, material properties and the amount of reinforcement materials, and the presence of

impurities on the corrosion properties of the metal matrix composites will be addressed. A particular focus is placed on the corrosion behavior of magnesium and aluminum metal matrix composites.

COMPOSITES: TERMINOLOGY AND TYPES

Composites can be defined as "A material consisting of two or more distinct phases bonded together" [2]. Composites are made of a matrix material, a homogenous and monolithic material in which a reinforcement is embedded. Composites are classified into Metal Matrix Composites (MMCs), Polymer Matrix Composites (PMCs), Ceramic Matrix Composites (CMCs), and Carbon Matrix Composites based on the matrix material (Metal, Polymer, Ceramic, and Carbon) used. Typically, composites are designed so as to offer remarkable mechanical and physical properties, making them one of the most significant advances in materials development. Classification of matrix composites is done based on matrix material, the structure of reinforcements, the type of reinforcement, and the orientation of the reinforcement see Table **1**.

Table 1. Classification of composites [3, 4].

Matrix Material	Structure of Reinforcement	Alignment	Reinforcement Material
Metals	Particulate	Continuous	Oxides
Ceramics	Structural	Discontinuous	Carbides
Polymers	Fiber	Randomly Aligned	Nitrides
Carbon	Powder	Interconnected	Borides

Polymer Matrix Composites

Polymer matrix composites are the most widely used matrix composites due to their low manufacturing costs and versatility. The general properties displayed by polymer matrix composites are low-strength, low-modulus, low density, resistance to corrosion, and good formability.

The properties of composite materials depend on the type of reinforcement (*e.g.*, particle, continuous fibers, discontinuous fibers, whiskers) see Fig. (**1**), chemical composition of reinforcement (*e.g.*, ceramics, metals, and carbon), orientation of reinforcement, amount of the reinforcement and the processing method adopted. As polymer matrix composites combine polymers typically with ceramics, their chemical and physical properties and fracture mechanisms differ significantly from either of their constituent materials. Most of the polymer matrix composites are usually anisotropic, especially those with continuous fiber reinforcements. Composites containing equiaxed reinforcement particles can be considered

CHAPTER 9

Aluminium Nanocomposites Developed by Additive Manufacturing for Use in Automobile Applications: Advances and Approaches

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Abstract: Aluminium has a lightweight (density is 2.7 g/cm^3), high specific strength, and excellent wear and corrosion resistance properties. Due to these properties, aluminium and its alloys are the most commonly used for structural, automobile, and aerospace applications. However, these monolithic materials have poor mechanical properties which are significant barriers to their further development. The resulting materials, when reinforced with ceramic particles, enhance the properties of materials and are capable of meeting the majority of industrial requirements. The reinforcement of ceramic affects the properties of developed composites. The composite fabricated by the conventional process has a limitation to the segregation of reinforced ceramic particles, porosity, weak interfacial bonding, and lower strength. Besides, additive manufacturing (AM) provides design freedom and dense and high-strength components. In the present study, advances in aluminium nanocomposite developed by laser powder bed fusion processes have been studied in detail. In addition, the objective of this chapter is to focus on the fabrication routes, formation mechanisms, effect of process parameters and its effect on laser absorption, grain refinement, interfacial bonding and mechanical properties of aluminium nanocomposite discussed in detail. The future scope of laser-processed aluminium composite is also briefly discussed.

Keywords: Aluminium composite, Additive manufacturing method, Classifications of additive manufacturing techniques, Laser powder bed fusion processes, Laser additive manufacturing, Microstructure, Mechanical properties.

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OVERVIEW

Aluminium is lightweight (density is 2.7 g/cm^3), and has high corrosion resistance due to the formation of a thin passive film on the exposed surface. Aluminium and its alloy are the most widely applied in the fields of construction materials, vehicles, aerospace, ship, *etc*. due to their high specific strength, light weight, excellent wear and corrosion resistance, ease of casting and lower thermal coefficient values [1 - 4]. In the present scenario, the industries demand environment-friendly, high strength lightweight materials, which have exceptional mechanical, wear, and corrosion resistance properties. Aluminium alloys are well-known materials for lightweight applications. However, these monolithic materials have poor mechanical and wear resistance properties which are significant barriers to their further development. The resulting materials, when reinforced with ceramic particles such as Silicon carbide (SiC), Titanium Dibromide (TiB$_2$), Aluminium oxide (Al$_2$O$_3$), Titanium carbide (TiC), Boron carbide (B$_4$C), and Titanium nitride (TiN), enhance the properties of materials and are capable of meeting the majority of industrial requirements [5 - 8]. Aluminium Silicon alloys (Al-Si alloys) are well-known materials for automobile moving parts such as pistons or cylinder heads in engineering components in automotive, aerospace and defence applications [9]. Silicon carbide and aluminium oxide reinforced aluminium composites are commonly utilised for automotive applications such as brake drums and cylinder liners, as well as rotor vanes and plates for structural aerospace parts [10, 11]. Though metallic composites may be developed by several techniques such as melting casting, and powder metallurgy processes [12]. However, these fabrication techniques have challenges such as the segregation of reinforced particles, weak interfacial bonding, non-uniform distribution, difficulty in machining, higher porosity and inferior mechanical properties [13]. Besides, additive manufacturing provides design freedom, intricate structure, and cost-effective, environment-friendly, dense, and high-strength components [13]. Furthermore, the reinforcement of ceramic particles in laser processing strengthened the interactions between the laser beam and the powder particles resulting in improving laser absorptivity, which helps to resolve the high laser reflectivity problem compared to monolithic aluminium alloy. In addition, the reinforcement of nanopowder in metal matrix material by laser powder bed fusion (L-PBF) process increases the laser absorption, improves the fluidity of the melt, refined grain structure, interfacial bonding, high-temperature performance, and higher mechanical properties [14, 15]. Additive manufacturing is the method of combining materials to form objects through layer by layer deposition of materials from specified three-dimensional model (3D model) data, and it is the process of creating the final shape by adding materials. The additive manufacturing process involves three basic steps, *i.e.*, (a) a computerised 3D solid model is created and converted into a standard additive manufacturing file format,

such as the traditional STL (standard tessellation language format); (b) the file is sent to an additive manufacturing machine and manipulated, such as changing the part's position and orientation or scaling; and (c) the part is built layer by layer on the additive manufacturing machine [16 - 18]. This process does not require cutting tools, coolants, fixtures, and other auxiliary resources for assembly [16].

In the present work, the methods and challenges associated with the fabrication of aluminium composite by additive manufacturing processing will be addressed. The additive manufacturing technologies are first introduced to provide a fundamental grasp of the additive manufacturing methodology. Following that, various fabrication processes, microstructure, and mechanical properties are discussed. The final section discusses the advantages, limits, and prospects of additively made metal matrix composites (MMCs). Finally, the key challenges in using metal additive manufacturing methods to fabricate metal matrix composites are examined and highlighted.

ADDITIVE MANUFACTURING METHOD

The additive manufacturing technique uses digital data from the computer to manufacture parts layer by layer from the lowermost up. Additive manufacturing is classified into seven techniques, *i.e.*, VAT Photo polymerisation, Binder Jetting (BJ), Sheet Lamination (SL), Material Jetting (MJ), Material Extrusion (ME), Powder Bed Fusion (PBF), and Directed Energy Deposition (DED), and these techniques are classified in terms of applied processes *i.e.*, direct or indirect processes [13, 17]. The flow diagram of additive manufacturing steps and the classifications of additive manufacturing techniques are shown in Figs. (**1** and **2**).
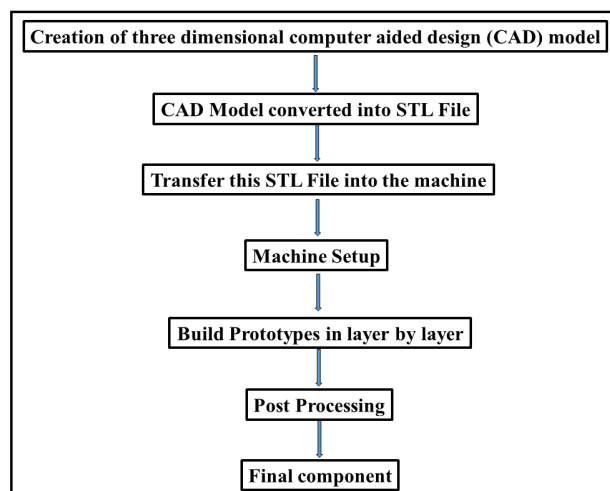

Fig. (1). Flow chart of steps involved in the fabrication of additive manufacturing components.

CHAPTER 10

Enhancing the Strength of Aluminum-Boron Carbide Composites to a High Degree by Magnesium Addition for Use in Automotive Applications

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Abstract: We report here a significant enhancement of the hardness of aluminum-boron carbide composites by the addition of magnesium. Reactive sintering between boron carbide and aluminum-magnesium occurs by the application of heat and pressure and during subsequent annealing at high-homologous temperatures of the matrix. In this case, the deformation-induced plastic yielding enables the incorporation and dispersion of hard particles in aluminum-matrix. We examine the decomposition behavior of boron carbide at high-homologous temperatures in contact with magnesium and aluminum, and observe the interfacial, aluminum-magnesium-boride, $AlMgB_4$, and aluminum-boro-carbide, Al_3BC, phases at boron carbide/matrix interfaces as revealed by the high-resolution transmission electron microscopy. We demonstrate that the hardness of these composites has been enhanced by two to five folds as compared to the base alloy and the existing aluminum-boron carbide composites. The addition of magnesium improves interfacial cohesion significantly between the matrix and ceramic particles as a result of interfacial boride phase, and primarily contributes to the enhancement of strength. This provides a novel method of developing aluminum-based high strength composites.

Keywords: Aluminum alloys, Boron carbide and transmission electron microscopy, Interfaces, Microstructure, Metal matrix composites.

INTRODUCTION

Metal matrix composites (MMCs) made of ceramic particles in metal matrix often exhibit improved hardness, toughness and wear resistance as compared to the base matrix without the particulates [1 - 8]. To lighten the weight and to increase the strength of composite materials, alumina (Al_2O_3) silicon carbide (SiC) and boron

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carbide particulates would be useful candidates. Aluminum alloys reinforced with boron carbide fabricated to produce the metal-matrix composites find applications in automotive [9, 10] in defense sector and in tribological sectors [11]. It is clear that much work has been done and continues to be done to improve many properties of these material especially the hardness of these materials for tribological and military applications. In the present work, we report a process by which the hardness of these composites has been enhanced by two to five folds as compared to the base alloy and the existing aluminum-boron carbide composites. It is a very significant result when compared to incremental improvements reported in the literature quoted above and also discussed below. In addition, we propose a mechanism by which this result has been achieved and provide transmission electron microscopy (TEM) observations in support of this mechanism.

The motivation here is thus to enhance the hardness and strength level of aluminum matrix boron carbide composites so that these composites can be useful for applications, where high specific strength or hardness is required. The maximum level of hardness observed in the present work is 400 VHN ($\approx$ 4GPa) for 12 wt.% boron carbide. Note the hardness of the existing aluminum-boron carbide composites, as mentioned before, is in the range of 120 to 140 VHN. We show for the first time the addition of magnesium to aluminum and boron carbide can improve the strength of the aluminum boron carbide composite enormously, which acts here as a binder and helps consolidate the composite at high temperature and pressure, by forming an interfacial aluminum-magnesium-boride phase between aluminum and boron carbide. In this case, the manufacturing process was conducted in the solid-state using ball milling and then by consolidating the powders at higher pressure and high homologous temperature of the matrix. We use transmission electron microscopy to investigate the interface phase formation in detail. As the consolidation process involves interfacial reaction between aluminum/magnesium and boron carbide, understanding the interface and defects at nano-scale and the fine scale microstructure is expected to be crucial to control the properties of these composites.

Several types of composite materials, such as multilayer composites, dispersion strengthened composites and fiber reinforced composites have emerged. Multilayers made of hard and soft layers result in higher strength and toughness [12 - 14] due to Koehler effect. It was argued in the multilayer composites if one of the individual layers has a significantly lower shear modulus, dislocations need to overcome a large repulsive image force to move from one layer of lower shear modulus into the other layer with higher modulus. On the other hand, in the dispersion strengthened composites, higher strength is achieved by impeding the motion of dislocations by hard particles dispersed within the matrix. In this case,

the load is carried away by the matrix. We recently reported in aluminum/alumina multilayers, the formation of nanocrystalline alumina within the soft layer, which enhances the overall hardness and strength of the multilayer composites by means of the Orowan strengthening mechanism [15].

Considerable work has been done to produce dispersion strengthened metal-matrix composites by adding various volume fractions of boron carbide in aluminum matrix [16 - 18]. These hard-ceramic particles are mostly added in liquid metal to form metal-matrix composites upon solidification [18]. For example, the aluminum 7475 - boron carbide composite was manufactured by using a stir casting method, and the boron carbide particulates were added into the molten metal. To achieve homogeneity, the mixture was stirred continuously by using a mechanical stirrer. However, this method tends to produce more inhomogeneity upon solidification of the composites, because the solid/liquid interface pushes hard-ceramic particles towards the end, which results in inhomogeneity in the solidified product. To achieve better homogeneity, aluminum-based metal-matrix composites were manufactured in the solid state reinforced with boron carbide particles [19 - 22]. In most cases, aluminum 7xxx, aluminum 6xxx and aluminum-2xxx series alloy powders along with boron carbide as a reinforcement have been used. However, all of these composites, manufactured in solid as well as in the liquid state, do not show better performance. As mentioned before, the enhancement of hardness or strength level is relatively small, 10 to 20%, as the bonding between aluminum-matrix and boron carbide is weak, and composites mostly fail as a result of de-bonding at the metal/ceramic interface. In these prior works, the enhanced hardness value has been reported to be in the range of 120 to 140 VHN for 8 to 10 wt.% boron carbide in aluminum [19 - 22].

Hence, to enhance the strength and hardness of the composites, one needs to improve the interfacial bonding between ceramic particulates and matrix. It has been realized a thin layer of a metal-boride phase at the interface could improve the adhesion between aluminum and boron carbide. In addition, these hard-ceramic particles need to be incorporated and finely dispersed within grains to impede the dislocation motion, so that the Orowan strengthening mechanism is operative [15, 23]. Not much work has been done to investigate the interfaces and the reaction layers at nanoscale formed at high homologous temperatures and high pressures. Recently, some work has been reported to improve the mechanical properties of pure magnesium and its alloys [24] with nickel and copper. They reported that the improvement of strength in pure magnesium [25, 26] is due to the presence of intermetallic phases, such as Mg_2Ni and Mg_2Cu in the matrix.

Processing and Fabrication of Sisal Fibers Reinforced Composites: A Conceptual Review

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Abstract: This study provides a short, succinct and convincing review of the studies researchers have performed on sisal natural fibre and its applicability for selection and use as a potentially viable and economically affordable reinforcement for engineering composite materials. As the technologies are developing rapidly day by day and the demand for developing and manufacturing eco-friendly materials is also increasing, the use of the bio-degradable reinforcements, like bamboo, sisal, jute, cotton and hemp, is of utmost concern. Made evident from a few to several other research studies, in comparison to the fabricated natural fibre-reinforced composite materials, the sisal fibres indicate considerable results that favour their applicability. Environmental contamination happens in various aspects of industrial processes, such as production, disposal, and recycling of synthetic fiber-reinforced composites due to their widespread selection and use. Sisal fiber is a good example of an environmentally acceptable natural fiber having significant mechanical qualities, which can be utilized for reinforcing a variety of polymer matrices. This review article provides a coverage of the intricacies specific to the manufacture of sisal fiber-reinforced composite materials, variables that impact their characteristics, surface treatments used to prevent the presence of flaws, and mechanical tests required to determine their strength. Prior testing attempts on the sisal fiber-reinforced composites are addressed in order to aid in future research to reveal the advantages and usage of such a material in a more effective and efficient manner. The current era of rapidly changing manufacturing environment and technological advancements highlighting such scenarios calls for a substantial amount of research to be performed on: (i) structure of sisal fibre, (ii) sisal fibre extraction/preparatory processes, and (iii) surface treatments of sisal fibres combined with other matrices.

Keywords: Mechanical properties, Mechanical testing, Natural fibers, Natural composites, Preparation process, Sisal fiber, Surface treatments, Sisal fiber polymer matrix composites.

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INTRODUCTION

Composites exist everywhere in the environment. Long cellulose fibers are bound together by lignin in a piece of wood, resulting itself in a composite. With the rising environmental concerns, the demand for developing eco-friendly and sustainable materials has exponentially increased. The bio-composites incorporating lignocellulosic fibers as a reinforcement in a polymer matrix are thereby considered as an effective and sustainable solution to the rising plastic waste. Composite materials are generally composed of two or more materials, which have significantly different properties. However, they do not react or merge together and this actively demonstrates that their properties work along in a concert. The composite's many ingredients combine to give it its distinct properties. Humans have employed composite materials in a variety of purposes for thousands of years. Around 1500 BC, the Egyptians and Mesopotamians used a mix of mud and straw to construct sturdy and long-lasting constructions. The combination of mud and straw within a brick provides exceptional compression, tearing resistance and bending resistance. Medieval composite items, such as ceramics and boats, remained to be reinforced with straw. The interconnections of different constituents, which makes it possible for composites to exist are shown in Fig. (**1**). Taking into account the functioning of composites, several functions that are performed by two key components – matrix materials and reinforcement materials have been addressed accordingly.
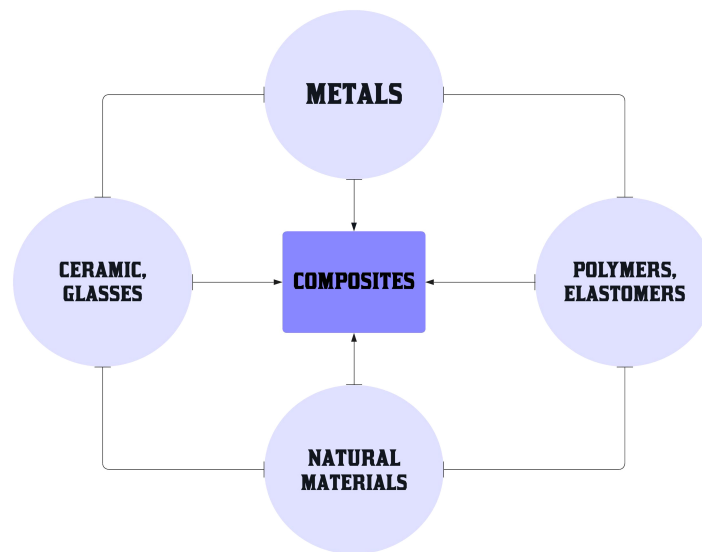

Fig. (1). Various classes of composites.

Matrix (Primary) Phase

Matrix phase is used to produce overall bulk form of a composite material component or product. It keeps the embedded phase, or reinforcements, *in situ*, which means by enclosing and hiding it. Whenever a load is employed, the matrix phase allocates its burden to the reinforcing phase. However, it may deform in some situations, allowing the reinforcing phase to bear the full load.

Reinforcing (Secondary) Phase

Their purpose is to support the primary phase. The most typical forms of embedded phase are fibers, particles, and flakes.

Fig. (2). Usage of natural reinforced fiber composites (NFC) Work in actual scenario [Reference 2].

NATURAL FIBER-REINFORCED COMPOSITE

Previously, the use of synthetic fiber composites was booming. Soon countries around the globe started to acknowledge facts about pollution issues and how to prevent them. Then came the era of green techniques, which were created to

On the Selection and use of Biomaterials and Implants in Orthopedics: A Study and Evaluation into the Future

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Abstract: The selection of suitable biomaterials and implants is the most important criterion to achieve success in biomedical engineering. Several factors such as high specific strength, high corrosion resistance, enhanced wear resistance, biodegradability, and biocompatibility need to be considered before choosing the material for biomedical applications. The biomaterials are developed from metals, metal alloys, ceramics, and polymers based on specific applications. In the orthopedic field, inert materials have been used in earlier times that showed minimal cell-material interaction, and bioinert materials were preferred to avoid immune rejection and increase the success rate. Metallic alloys such as Ti-6Al-4V, 316 stainless steel, and cobalt-chromium are frequently used in the fabrication of bone implants. Metal alloys have problems like corrosion and are less bioactive. In recent years, the concept of regeneration has been developed and it is entirely based on cell-material interactions, and the selected materials need to exert bioactivity. Regenerative materials are prepared from biodegradable metals, polymers, and ceramics. Numerous techniques have been developed recently to convert a bioinert material into bioactive or regenerative material. Smart materials and implants with bioactive surfaces are also getting more attention in the orthopedic field. This chapter throws light on two fields that have contributed to bone replacement and regeneration. An overview of the bioinert implants in the orthopedic industry and the current development of bioinert into bioactive and immunomodulating materials will be discussed. The future aspects describe the role of smart materials for bone regeneration in detail.

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Keywords: Additive manufacturing, Biodegradable implants, Cobalt-chromium, Orthopedic implants, Smart implants, Surface modifications, Surface texturing, Stainless steel, Ti-6Al-4V.

INTRODUCTION

Bone trauma and fractures affect the lives of many people all around the world [1, 2]. Autografts and allografts have been used for treating small defects [3]. In case of larger defects, auto or allografts are not possible and bone grafting materials or implants came into the role. Orthopedic implants are divided into two categories namely load-bearing and non-load-bearing implants based on their applications. Non-load-bearing implants are used to provide structural support but not to withstand the load. These non-load-bearing implants are made up of ceramics and biodegradable polymers. However, load-bearing implants must hold the weight of the patient, and the structure of metal alloys. These types of implants are used in artificial knees and hip joints, where the action of load will be high [4 - 8]. In the 19th century, metals and their alloys have been used extensively as medical implants. As the economy and technology advanced, industries started to produce a huge variety of metals and alloys. Few metals and alloys are biocompatible and have a proven track record of effectiveness in medical implants [9].

Metal and metallic alloys have unique surface properties, high mechanical strength, biological response, and heat transduction characteristics. Due to the high mechanical properties and fracture toughness, metal implants were used in load-bearing applications. Pure metals easily corrode and have not shown promising results in orthopedics [10, 11]. Metallic Bio-implants such as titanium and its alloys [12], stainless steel [13], and cobalt-chromium alloys [14] are widely used in orthopedic applications. The above-mentioned alloys show controllable corrosion rates and have greater strength in bone implant applications. Titanium (Ti) alloys have been most commonly used in orthopedics for more than 50 years. Titanium alloys consist of different structures such as equiaxed, bi-model, and laminar, which show high ductility, strength, and creep resistance properties, respectively. The most studied Ti-alloy is Ti-6Al-4V (Ti-Titanium, Al-Aluminum, V-Vanadium) (Grade 5 titanium), which is reported to have low density, a high strength-to-weight ratio, and very good biocompatibility. The presence of 6% Aluminum (α phase) and 4% of Vanadium (β phase) stabilizes the α+β phase of titanium and helps in achieving high strength and ductility properties. It is also reported that the oxide layer formed in this alloy enhances the adherence of bone progenitor cells. Stainless steel (SS) has been the choice of implant material among all other alloys due to its low cost and easy production. The chemical composition of stainless steel contains 18% of chromium, which helps form the chromium oxide (Cr_2O_3) layer on the surface of

materials. The oxide layer further enhances the corrosion properties and biocompatibility of materials. However, the high stiffness of the Stainless steel-based implants leads to high bone resorption, and these implants are reported to induce inflammatory response at the site of implantation. Cobalt-chromium is one of the high-hardness materials, which is used in load-bearing applications such as hip and knee joints. Hence, the material predominately requires high surface hardness and tribological resistance to avoid wear debris formation. The chemical composition consists of 27-30% Chromium and 5-7% Molybdenum. This composition helps to attain a homogenous microstructure and high tribological resistance compared to other metallic implants. Considering the corrosion, and less biological response of conventional metal alloy implants, researchers are focusing on the development of biodegradable and bioactive implants. Magnesium-based bone implants are getting more interest in the area of biodegradable implants. Magnesium has high osteoconductive properties and very good biocompatibility. It can also be used in load-bearing applications [15]. The high corrosion behavior of magnesium-based implants needs to be controlled for their successful application in orthopedics.

There is a need to keep several points in our mind before selecting a material for bone implant applications. Many reasons can be attributed to the failure of implants. The main reason behind the implant failure is the properties like corrosion, strength, and biocompatibility of the material selected [16]. The other reason could be a failure due to the infection in the implants. Stress-shielding effect of the implants is another major reason for implant failure at the site. Bone remodeling maintains the external and internal structure of the bone in response to the mechanical forces that occur during a normal biological response. The remodeling process maintains the strength of a bone by changing the calcified tissue properties in response to the loading conditions met by the skeleton. The bone density and geometry will be maintained by osteoblasts (bone-forming cells), and osteoclasts (bone-resorbing cells). These cells will be activated by the biological systems in response to the mechanical forces. When bone implants have high stiffness, it would alter the distribution of load in the bone. The stress-shielding effect may reduce the load that is present in the bone previously and that leads to poor remodeling [17, 18]. Due to the poor remodeling, the shielded bone may have a lower density with a more porous structure and anatomically smaller in size. Further, due to the high stiffness of the implants, the load can act in different areas which are never exposed to high loads. This may result in bone damage and fracture. This abnormal stress concentration is also a reason for implant loosening and failure. To prevent the stress-shielding effect of the implants, the geometry should be designed accurately which will allow the proper distribution of load throughout the implant.

Smart Hydrogels: Theory and Applications with Particular Focus on Biomedical Sciences

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Abstract: Smart and intelligent materials provide direct or indirect solutions for many of the current/existing problems. Studies in this line have been in progress since few decades developing multifunctional materials, smart systems *etc*., for specific applications. Though many smart materials were developed, not much of them are compatible as implants in human body. One such material that possesses the inherent requirements attuned with human body is 'hydrogel'. Like other smart materials, gels can also be intelligent in their responses to the environments they are exposed to, or on influence of any induced stimuli. Many such smart gels are explored by various industries and are used for applications such as drug delivery systems, cancer therapy, tissue engineering and organ printing, but a comprehensive understanding with properties, and their mechanisms regarding their functionality and usage are meagre. This work consolidates and details the classification of gels and elaborates the various stimuli to which gels respond and the mechanisms involved. This article also explores the possibilities, prospects of 3D-printing of smart gels in biomedical field and has listed the possible applications of hydrogels.

Keywords: 4D printing, Additive manufacturing, Artificial lens, Artificial muscles, Bimodal, Biochemical, Cross-linking, Drug delivery, Hydrogels, Hydrophilic, Hydrophobic, Organ printing, Self-healing, Shape memory hydrogels, Smart gels, Spinodal, Stimuli responsive, Superporous, Supramolecular, Tissue engineering.

OVERVIEW

Progress in materials science requires the development of newer materials and systems to cater to the needs of the ever-demanding applications. The advent of multi-functional materials has benefited the scientific community in building up systems/technologies to provide minimally-invasive surgeries, smart diagnosis,

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organ printing, *etc*. Such smarter systems have transformed dumb machines/systems to sophisticated ones that provide better methods for easy diagnosis and cure. Smart/Intelligent materials are the key to the development of such smarter systems that exhibit the capability of sensing, judging, and responding to environmental conditions optimally, thereby contributing to the evolution of smarter technologies for the betterment of civilisation. Stimuli response is one of the basic phenomena in nature that offers comprehensive avenues for learning and the same can be adapted to the material systems for providing solutions to many of the current problems [1]. Smart materials possess the characteristics of changing their properties on subjecting to an external stimulus and the external stimuli to which these materials respond can either be pressure, moisture, pH, presence of chemicals, light, temperature, electric or magnetic fields or a combination thereof. Gels are one such smart material that can respond to a variety of stimulus and are capable of providing specific functions in the human body. Irrespective of the various fields of application, science and engineering of gels open a larger domain in the bio-materials world.
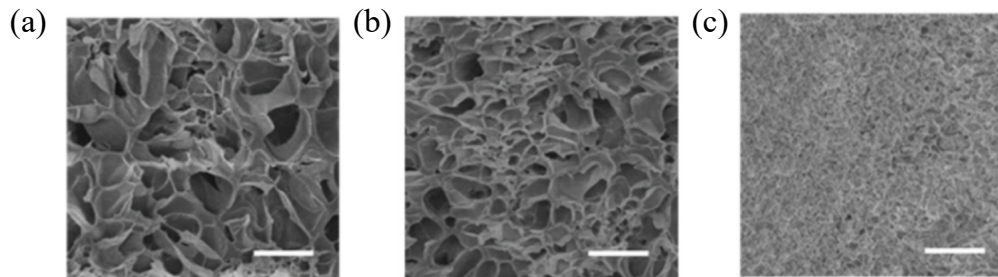
Fig. (1). SEM micrographs of PAAm-CS hydrogel; (**a**) With solvent, not exposed to stimuli; (**b**) Soaked in 0.75 mM Sodium Hydroxide; (**c**) Soaked in saturated Sodium chloride; all micrographs with scale bar indicating 10 μm (Reproduced with permission from Xiao, He. Ma, Chunxin. Le, Xiaoxia. *et al*., A Multiple shape memory hydrogel induced by reversible physical interactions at ambient condition. Polymers. 2017; 9, 138, with permission).

The challenges posed by the biomedical field required the development of lighter and biocompatible materials for drug delivery and other biological applications and hence an investigation of gels became more pertinent. Biodegradability and biocompatibility were the challenges for plastics in the fields of minimal invasive surgery, synthetic degradable implants, suture materials and organ printing, for which gels offers a ready and immediate solution [2]. Hence, it will be interesting to study and consolidate the nature and variety of gels along with exploration of the smart actuation capabilities with their mechanisms and applications as a ready-reference to the scientific community.

GELS, THE WONDER MATERIAL

Gel, Sol, and Emulsion are three different states of colloidal solutions, which differ in the dispersion phase and medium. While emulsion represents a colloid in which the dispersion medium and dispersion phase are liquid (example: Paints), sol represents the dispersion of solids in a liquid medium and gels represent the dispersion of the liquid phase in a solid medium. The solid medium in a gel is analogous to the sea sponge, an ocean creature with its body full of channels and interconnected pores permitting water to percolate. Transformations between sol and gel depends on the cross-link stability and are being studied across the globe using thermodynamic concepts, combining the advantages of each and to develop smarter systems. The ability to transform between gel and sol, in response to certain stimuli, due to the absence of strong cross-linking, makes gel a favourable material for use in the biomedical field [2 - 4]. Gels are classified as a category of soft and wet materials with three-dimensional cross-linked networks in a large porous solvent matrix and it possesses the characteristics of high fluid absorption that results in the shape memory behaviour. The inherent nature of gels makes it possible to accommodate fluids in the pores and cause a change in volume (by swelling) and also withdraw the fluid to regain its original volume attributing to a change in shape. Gels that can absorb water are termed as hydrogels and are generally composed of hydrophilic polymer chains that have affinity towards water. The hydrophilic groups like amino, carboxyl and hydroxyl groups in the polymer chains are responsible for the water holding ability of the hydrogels that results in the change of volume by means of swelling [5 - 8]. Gels also exhibit extremely low friction coefficient and are regarded as soft and high ductile materials with prolific applications that include lubricants for aerospace materials, templates for nanoscale and other biomedical devices, scaffolds for tissue engineered prostheses, lens, eyeball, artificial muscles, artificial blood vessels, smart buttons, biosensors and actuators [9, 10].

When liquid phase of the gels is composed of organic solvents in polymeric network instead of water (as in hydrogel), they are termed as organogels. These organogels are usually non-crystalline, non-glassy and thermoplastic in nature. A polymeric hydrogel gel, which is devoid of water, is called xerogel, and the amount of water that can be absorbed by a hydrogel may vary between 0.1 and 1000 of times of the weight of the xerogel [4, 9]. Apart from these three types of gels, a hybrid variety of nanocomposite hydrogels are possible that are either physically or covalently cross-linked and has nanoparticles (carbon-based, polymeric, ceramic, metallic) embedded in the matrix those enhances the microstructure. These hydrogels are capable of mimicking human tissues, structure and microenvironment due to highly hydrated and interlinked pores [10].

Engineered Iron-Oxide Based Nanomaterials for Magnetic Hyperthermia

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Abstract: Ferrite nanomaterials are extensively studied for their use in the biomedical field primarily because of their tunable magnetic properties and biocompatibility. The use of magnetic nanomaterials, particularly the iron-based nanoparticles, for hyperthermia treatment is one of the emerging applications. However, there are practical constraints on the overall applicability of pure iron-oxide nanoparticles (IONPs) for hyperthermia treatment. In this regard, doping foreign metal ions in the crystal lattice of pure iron-oxide nanoparticles (IONPs) possessing a spinel or inverse-spinel structure remains to be the simplest approach for the purpose of improving the desired properties. Doping other metal ions into the iron-oxide nanoparticles (IONPs) causes strain in the crystal lattice and is responsible for engineering the structural properties and magnetic properties. Various elements, such as the rare-earth (RE) metals, especially the lanthanides [Yttrium, Gadolinium and Europium], the transition metals [manganese, cobalt, nickel and zinc], and other metals [gold, silver, calcium, titanium, copper and magnesium] are being investigated for their potential to serve as dopants. The divalent transition metals [manganese, cobalt and nickel] doped iron-oxide nanoparticles possess highly improved magnetic properties. Incorporating trivalent ions of lanthanides improves the structural properties, magnetic properties, and dielectric properties of the iron-oxide nanoparticles (IONPs). Moreover, doping with zinc, gold and silver imparts the ion-oxide nanoparticles (IONPs) with antibacterial properties while concurrently tuning their structural properties and magnetic outputs.

Keywords: Doping, Iron-oxide nanoparticles, Lanthanides, Magnetic hyperthermia, Rare-earth metals.

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INTRODUCTION

The iron-oxide nanoparticles (IONPs) possess unique and tunable magnetic properties that enable them to find versatile applications in the fields ranging from electrical/electronics to nanomedicine. These nanoparticles (NPs) are also considered to be safe for the purpose of biomedical applications and thus can be used as nanocarriers for the following: (i) drug delivery, (ii) nano heaters in magnetic hyperthermia, and (iii) contrast agents in imaging [1 - 6]. The iron-oxide nanoparticles (IONPs) consisting of a single domain structure possess superparamagnetic character when the temperature is between the curie temperature (T_C) and the blocking temperature (T_B), *i.e.*, $T_C > T > T_B$. Upon exposure to an external magnetic field, these nanomaterials possess high magnetization. However, they tend to lose their magnetization as soon as an externally applied field is removed [7]. Typically, the iron-oxide nanoparticles (IONPs) below a critical size of 20 nm possess superparamagnetic properties and are termed as superparamagnetic iron-oxide nanoparticles (SPIONs) [8].

Among iron-oxide nanoparticles (IONPs), magnetite bearing chemical formula Fe_3O_4 received excellent attention from researchers and finds its use in a diverse number of applications. The Fe_3O_4 possesses an inverse spinel structure consisting of closely-packed oxygen ions with the cations occupying interstitial sites [9]. It is generally represented as AB_2O_4 where, A represents the tetrahedral sites occupied by the Fe^{3+} ions while B sites are the octahedral sites engaged alternatively by the Fe^{2+}/Fe^{3+} ions and thereby giving the formula $[Fe^{3+}]_A[Fe^{3+} Fe^{2+}]_B O^{2-}_4$. The Fe^{3+} ions have a magnetic moment of $5\mu_B$. However, Fe^{2+} ions located at the octahedral (B) sites contribute to the net magnetic moment of inverse-spinel ferrites *i.e.*, $4\mu_B$ [10]. This is essentially because of cancellation of the magnetic moment of Fe^{3+} ions being equally distributed among the tetrahedral sites and octahedral sites and having a magnetic configuration that is antiparallel to an externally applied magnetic field [11].

Factors like diameter/size, shape, geometry, and anisotropy of the iron-oxide nanoparticles (IONPs) exert an influence on their magnetic properties and heating abilities [6, 12 - 15]. Therefore, various strategies are currently being practiced by researchers to tune the magnetic properties of pure iron-oxide nanoparticles (IONPs). One such strategy is to dope with other metal ions into the crystal structure [16]. Doping refers to an intentional introduction of impurities in the crystal lattice of the pure iron-oxide nanoparticles (IONPs). The doped nanomaterials possess interesting magnetic properties, chemical properties, and dielectric properties. The addition of dopants tends to change the chemical order of pure ferrites. Therefore, different types of dopants added at varying concentration levels are currently with the intent of optimizing the electrical,

magnetic, and optical output of the pure iron-oxide nanoparticles [IONPs]. The various dopants used for developing engineered IONPs can be broadly classified as follows [7]:

(a) Transition metal-doped iron-oxide nanoparticles (IONPs),

(b) Rare earth (RE) or lanthanide-doped iron-oxide nanoparticles (IONPs), and

(c) Other metal-doped iron-oxide nanoparticles (IONPs).

Manganese (Mn), cobalt (Co), nickel (Ni), and zinc (Zn) among transition metals and europium (Eu), gadolinium (Gd), Tb, yttrium (Y), and Ho among lanthanides are commonly used to dope the iron-oxide nanoparticles (IONPs). Other metals ions, such as magnesium (Mg), calcium (Ca), gallium (Ga), strontium (Sr), barium (Ba), gold (Au), copper (Cu), and silver (Ag) also serve as dopant materials (Fig. **1**).
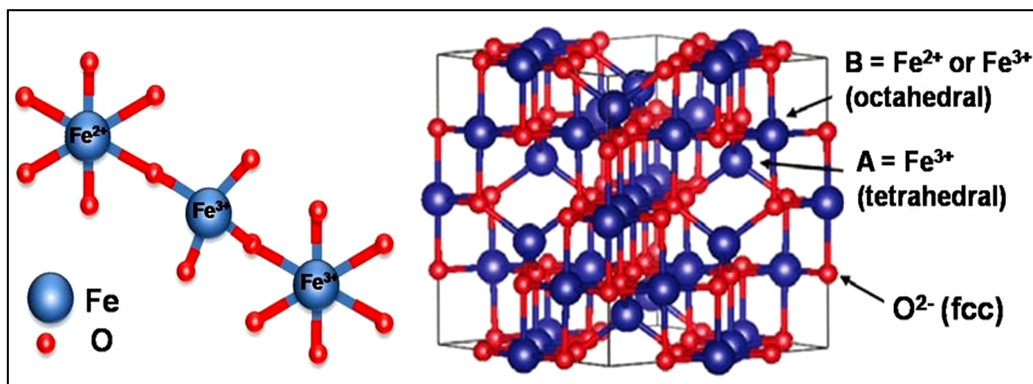
Fig. (1). The inverse spinel structure of Fe_3O_4 adapted with permission from rivani and co-workers [17].

The purity of crystal lattice, or chemical order, is an important factor which affects the physiochemical properties of the magnetite. It belongs to a space group *Fd-3m* that contains 32 oxygen ions (O^{2-}), which form face-centered cubic (FCC) unit with eight formula units *i.e.*, $Fe_{24}O_{32}$ with the cations stoichiometric (Fe^{3+}: Fe^{2+}) of 2 [18]. Interestingly, the oxygen framework provides flexibility to substitute Fe ions with other metal ions or cations, such as manganese (Mn), magnesium (Mg), nickel (Ni), cobalt (Co), copper (Cu), zinc (Zn), and gadolinium (Gd). The various elements explored as dopants for the iron-oxide nanoparticles (IONS) are listed in Table **1**. Doping of foreign cations alters both the physiological, and magnetic output of the iron-oxide nanoparticles (IONPs) by changing their magnetic orientation [19]. However, saturation magnetization of the doped system decreases when compared to the bulk magnetite, which

Emerging and Sustainable Material Technology: The Future of Fire Safety

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Abstract: Residential, workplace, commercial & wildfires claim and injure millions of people and cause trillions of dollars worth of property damage worldwide, considering both natural & man-made resources. The current review paper provides a clean, clear, cohesively complete and convincing review of the emergence of new and sustainable emergent material additives that improve fire-retardant properties and thermal stability, as well as their applications for the safety and protection of life and property in buildings. Globally emerging materials have entered the realms of construction, protection of critical apparatus, and self-protection equipment, thereby revolutionizing the level of fire protection for both people and materials. However, cost, application difficulty, mass-scale availability, and a lack of knowledge have frequently led to a lack of focus on preventing this calamity. In addition, emerging materials technologies can contribute to sustainable development by considerably enhancing the fire-retardant properties of recyclable waste, including both biological waste and industrial waste.

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INTRODUCTION

Throughout history, fire has been a companion to human beings and has played a pivotal role in their evolution. The identification of fire led to human settlement and the gradual formation of societies, with the agrarian economy taking the lead prior to the industrial revolution [1]. However, its widespread use did bring about numerous dangers. Now, fire risk and its dangers pose a substantial threat to both life and property [2]. Furthermore, polymeric materials are being increasingly selected and used in building structures due to their light weight, cost savings and low density. This makes polymeric materials quite easy for the purpose of both handling and processability while concurrently enhancing its demand due to aesthetic appeal. However, due to their poor fire resistance characteristics, the polymer-based building materials chosen for use in construction for insulation works, false ceilings, fixtures, and data cables are often prone to heat and resultant damage [3].

Despite its poor fire resistance characteristics, plastics became the most extensively chosen and used materials during the last few decades [4]. Between 1950 and 2012, the polymeric industry grew by 8.7 percent [5]. These multifunctional materials, have become indispensable for modern-day living, while also becoming vital to the modern economy. A detailed examination of the published literature reveals that the worldwide production of plastics has gradually increased to nearly 10 million metric tons during the last decade [6]. Now the world is also facing frequent catastrophic fire incidence due to the widespread use of flammable polymeric materials coupled with the rise in fire-related applications in human existence. These fires have frequently resulted in the loss of many lives.

In 2019, for instance, 1.2 million fire incidents occurred in the United States [7]. These incidents resulted in 3,704 fatalities and 16,600 injuries. The fire caused a net economic loss of approximately $14.8 billion. The European Fire-Retardant Association (EFRA) estimated that twelve fatalities and over one hundred twenty fire-related injuries occur daily. The World Health Organization (WHO) reports that on an average approximately 300,000 annual fire-related fatalities occur worldwide.

European fire safety alliance is reported to be close to 5000 deaths due to fire incidents every year [8], and the numbers are only showing an upward trend. These widely utilized plastics due to their unique chemical configuration and molecular structure have intrinsic fire prone characteristics, which frequently restrict their selection and use in both the manufacturing sector and construction sector.

Therefore, plastics exposed to a high temperature tends to decompose and lose their carbon bonds. It emits volatile gases that have a propensity to oxidize in air and form an ignitable mixture. This mixture has the tendency to ignite readily when exposed to an elevated temperature. However, if the polymer undergoes pyrolysis, it will sustain the continuous exposure to heat and the material will eventually get burned out and the flame will get extinguished. The combustion process will produce a mixture of toxic smoke, which is harmful to both health and the environment [9].

For the overall fire safety of structures, structural engineers have generally placed a greater emphasis on design intricacies and preferred the use of robust materials. In a short period of time, plastic fire fuels can significantly increase the temperature of a structure. This highlights the importance of preventing plastic fires and the required mitigation techniques to provide safety both to the material and the structure. Therefore, there is a need to integrate flame retardant compounds into the plastics that are chosen for use in such industries. It will decrease their peak heat release rate and ignitability.

The addition of flame retardants will increase the escape time during a fire. This could result in saving human lives. They are added to plastics in a variety of applications that require enhanced fire resistance. A vast majority of the plastics used in common applications have inadequate inherent flame resistance characteristics and therefore this necessitates the need for flame retardant additives. The total requirement for flame retardants in the European market exceeded 600,000 tons in 2015, with applications for use in the following: (i) transportation, (ii) construction, (iii) electrical and electronic equipment, and (iv) furniture [10].

The flame retardants delay a polymer's combustion in the event of a fire. Nevertheless, not all of them are secure for the environment. The latest regulation on halogenated flame retardants prompted a shift towards developing sustainable flame retardants. All organizations are currently working ondeveloping flame retardants that are sustainable, biodegradable, and kind to the environment. Additionally, a large number of countries have now made a commitment to set aside a portion of their GDP to combat climate change by lowering the emission of carbon. More governments are now supporting materials science and engineering research facilities to create sustainable polymers while concurrently abandoning halogen-based flame retardants [11]. Therefore, bio-based flame retardants, industrial waste-based flame retardants, and recyclable flame retardants quickly emerged as obvious candidates for this application. However, they need modification of both the physical properties and chemical properties of the material to have sufficient resistance to both fire and heat.

Recent Advances in Unconventional Machining of Smart Alloys for use in Critical Manufacturing Sectors

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Abstract: Nowadays, numerous metallic alloys are known to exhibit smart behavior; these metallic materials are categorized as shape memory alloys (SMAs). Shape memory alloys belong to a group of smart alloys that have the potential to recover their original shape from a deformed shape when triggered by factors, such as heat, electricity, and stress. This shape change gives rise to SMART behavior (Stimulated Martensite - Austenite Reverse Transformation). Shape memory alloys have attractive characteristics, such as high recovery force, high strength, *etc*. Shape memory alloys find their use in many applications *i.e*., aerospace, biomedical, automobile, robotics, *etc*. The ever-increasing demand for shape memory alloys among industries is attributed to their ability to respond to different machining processes. Conventional machining processes (CM) and unconventional machining (UCM) processes are the two major types of processes upon which the studies on shape memory alloys have been carried out. The machining studies carried out reveal that the use of conventional machining to process shape memory alloys to various products is considered undesirable since it causes damage and introduces changes in the characteristics of the material. Unconventional machining processes are therefore preferred. Various types of unconventional machining processes like laser beam machining (LBM), electrodischarge machining (EDM), electrochemical machining (ECM), abrasive jet machining (AJM), abrasive water-jet machining (AWJM), *etc*., generally involve machining of shape memory alloys in an unconventional way so that the wear rate and surface roughness are reduced. A study of unconventional machining processes is therefore considered essential contributing further to the domain of smart materials. Hence, through this review, the mechanism of shape memory alloys and their applications, various types of unconventional machining processes, and their recent advances are highlighted.

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Keywords: Abrasive water jet machining, Electrochemical machining, Electrodischarge machining, Laser beam machining, Martensitic transformation, Superelasticity, Shape memory alloys, Shape memory effect.

INTRODUCTION

The growth of technology, together with an increase in population, necessitates the requirement of smart solutions to real-world problems. One such smart solution is to use smarter materials to solve real-time challenges faced by different industrial sectors like medicine, manufacturing, materials, electronics, software, *etc.*, where the materials possessing better electrical properties, magnetic properties and other such characteristic properties are conspicuously absent [1]. One such class of materials that is trending in the market space is shape memory alloys (SMAs). Shape memory alloys were initially discovered and developed by Arne Olander in 1932, and it was in 1941 that shape memory was first described . The significance of the shape memory alloys was not acknowledged until William Buehler and Frederick Wang demonstrated the shape memory effect in nickel-titanium (NiTi) alloy [2 - 4]. Shape memory alloys are a class of intelligent materials which are capable of transforming into their undeformed original shape from the initial shape when they are subjected to external stimuli such as heat, stress, electricity, magnetic field, and so on [5]. The characteristics of shape memory alloys are given in Fig. (**1**). The two important thermomechanical properties that induce smart features in them are superelasticity (SE) and shape memory effect (SME) [6]. The occurrence of the shape memory effect is because of the martensitic phase transformation and its reversion to austenite. Technically, when an alloy is deformed in martensitic condition, it transforms to its original shape by following a reverse transformation, changing its structure from martensite to austenite (parent phase) [7].

Mechanism Overview of Shape Memory Alloys

Shape memory alloys demonstrate two kinds of phases possessing three non-identical crystal structures, namely twinned martensite and, detwinned martensite, and austenite. Apart from these phases with specific crystal structures, they show six possible types of transformation, as shown in Fig. (**2**). From the figure, it can be identified that at lower temperatures, the martensitic phase becomes stabilized, whereas at higher temperatures, the austenitic phase prevails. This is because when they are heated to a higher temperature, they change their shape and return to their original shape, *i.e.*, they transform from martensite (deformed structure) to austenite (parent phase) as the material is heated. This particular transformation that is taking place is referred to as martensitic phase transformation [8, 9]. In general, martensitic phase transformation is a solid-state diffusion less shear

dominant process involving nucleation of the crystal structure of the alloy that is responsible for the phase change from martensite to austenite in the alloys. This transformation is possible only if the total energy of the martensitic phase (product phase) is lesser than the total energy of the austenite phase (parent phase) at temperatures below the critical temperatures. Four distinct temperatures are used to indicate the start and end of the martensitic phase transformation processes. When shape memory alloys are cooled and deformed, there is a formation of martensite from the parent phase, and it starts to appear at Ms (Martensitic start temperature) and finally forms at Mf (Martensitic finish temperature). This transformation is particularly not seen because the martensitic plates self-accommodate themselves, leading to zero martensitic deformation. Similarly, when they are heated, there is a formation of austenite from martensite, and this initially appears at A_s (Austenite Start temperature), and it finally forms at A_f (Austenite Finish temperature) [10 - 13]. The mechanism of temperature changes is shown in Fig. (**3**). There is an intermediate temperature visible from the figure and this is termed M_d (Martensitic deformation temperature). It is the maximum temperature at which no further stress can be induced into the martensitic structure, and if the temperature reaches above this value, then the shape memory alloys start to plastically deform, similar to other metallic materials, and also, they lose their shape memory properties. The shape memory alloys are sometimes described as "functional intermetallic" because of the shape memory effect and super elasticity. The shape memory effect is the effect obtained when shape memory alloys respond to temperatures, and super elasticity is the phenomenon when shape memory alloys respond to applied stress. Both super elasticity and shape memory effect are categorized into three shape memory features as follows:

One-way Shape Memory Effect (OWSME)

It is the effect seen in the shape memory alloys only once, typically changing its shape to its original form only once from the deformed state upon removing the external load and exposing the alloy to higher temperatures.

Two-way Shape Memory Effect (TWSME)

Also known as the reversible shape memory effect seen in shape memory alloys that have the capability to retain their original shape both under high and low temperatures. This two-way shape memory effect is a process that can be repeated indefinitely as many times in the material.

Pseudo Elasticity (PE)

Also known as superelasticity (SE), it is a type of shape change effect when the

Critical Parameters Influencing High-Strain Rate Deformation of Materials Using the Split-Hopkinson Pressure Bar Apparatus: A Review

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Abstract: Strength and ductility of materials at high strain rate of deformation are important for design engineers working in a wide range of industries, especially in gas turbine engine blades. Split Hopkinson pressure bar (SHPB) apparatus is a popular method of characterizing the high strain rate behaviour of materials. The results from the split-Hokinson pressure bar tests are further used in mathematical models such as Johnson-Cook model, Zerelli - Armstrong model and so on for correlation purposes. Split-Hopkinson pressure bar tests take place in a very short duration of time (less than a second) but require significant effort in arranging the apparatus (several hours to days) and replications could be a daunting task. The present review work focuses on some of the critical test parameters that influence the test results. Specimen dimensions, its alignment in the split-Hopkinson pressure bar apparatus and location of strain gauges are critical parameters to be checked before the test (pre-testing) while selecting the accurate signal data processing technique (post-testing) to filter the noise which is critical to get meaningful test results. This review work focuses on the effect of three pre-testing parameters and signal data processing techniques on the high strain rate test results and summarizes the salient findings.

Keywords: Filter analysis, High strain rate behaviour, Johnson-cook model, Pulse shaper, Split hopkinson pressure bar, Signal data processing.

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INTRODUCTION

Engineering materials may be subjected to quasi-static and dynamic loading depending upon their applications. Behaviour of materials under dynamic loading is a matter of serious interest in applications such as aircraft, jet engines, nuclear reactors and so on. Impulse loading is a variant of dynamic loading where the load acts for a "very short" duration of time. Applications involving impulse loading such as in case of bird impact, crash, Foreign Object Damage (FOD), explosion, *etc*., result in a change in material stiffness due to the high strain rate of loading. Material response (behaviour) varies with loading rate and is an important design parameter to consider.

The most common method of determining the high strain rate behaviour of materials is by using a split-Hopkinson pressure bar (SHPB) apparatus. It works on the principle of one-dimensional wave propagation for a stress-coupled setup. It consists of striker, incident, and transmission bars, which are made of similar material, usually maraging steel, as shown in Fig. (**1**) [1]. The specimen to be tested is placed between the incident and transmission bar, while a heating furnace can also be used for testing the specimen at elevated temperatures. Usually, the high strain rate test occurs at $10^2 - 10^4$ s^{-1}, which is very high compared to quasi-static testing methods ($10^{-2} - 10^{-3}$ s^{-1}) [2].
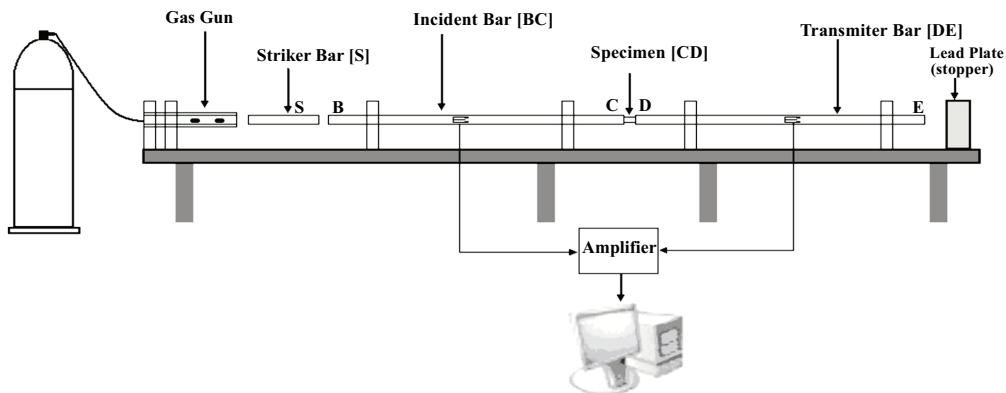
Fig. (1). Schematic of split hopkinson pressure bar [1].

During split-Hopkinson pressure bar testing, strain gauges are attached to the incident and transmitted bars to measure the deformation due to impact. Initially, a high-pressure inert gas from the compressor is used to drive the split-Hopkinson pressure bar test. Instant release of pressurized gas from the chamber pushes the striker bar forward at a high velocity. As depicted in Fig. (**1**), the striker bar

collides with the incident bar, causing stress waves, also known as incident waves, to be transmitted to the specimen mounted between the incident and transmitted bars. The specimen undergoes deformation by absorbing part of the incident stress waves while the remaining waves are transmitted through the sample to the transmitted and reflected bar at the interface, and are called transmitted and reflected waves, respectively [3].

These strain waves are captured using strain gauges, attached to the incident and transmission bar, and sent to the Data Acquisition Control (DAC) System. The raw data, in terms of load-deflection, is captured by the data acquisition control system, as shown in Fig. (**2**). It contains high-frequency noise as output, which cannot be directly implemented in any mathematical or analytical model for the analysis of the results. The process of filtering the noise from the data is called Signal Data Processing (SDP) which plays an important role in obtaining reliable and accurate test results from the experimentation.
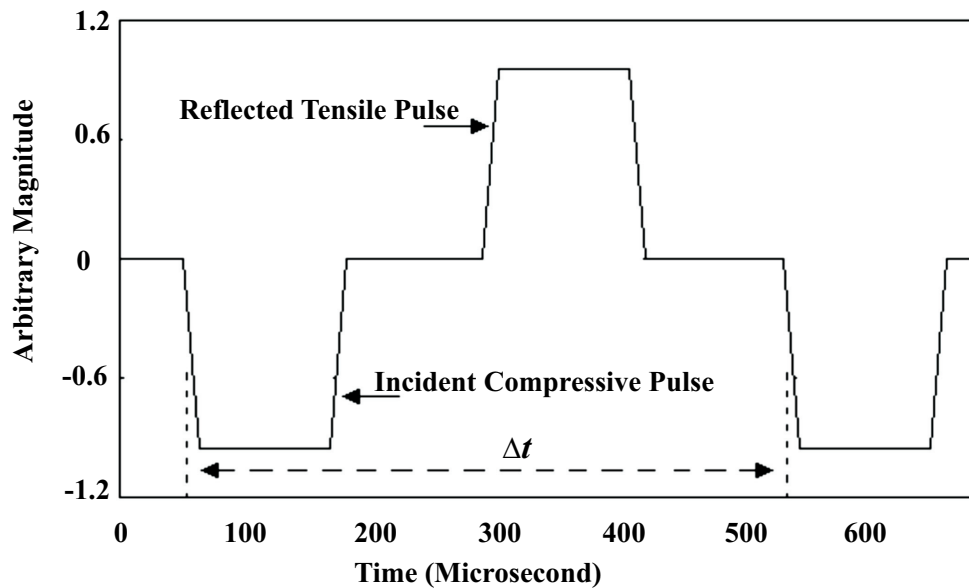
Fig. (2). Raw data showing different wave signals captured during the split-Hopkinson test [3].

The output of the split-Hopkinson pressure bar test is sent to the data acquisition system, and its function is depicted using a flowchart, as shown in Fig. (**3**). In the system, deformation signals that are in voltage values are sent to a rectifier through an amplifier where data processing occurs, and the signals are sent to an oscilloscope where they are converted with respect to time and finally displayed into the required format such as load-displacement, true stress-true strain *etc*. The

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