

Periodicals of
Engineering and Natural Sciences

PEN – VOL. 1 NO. 1
(2013)

Periodicals of Engineering and Natural Sciences (PEN)

Periodicals of Engineering and Natural Sciences (ISSN: 2303-4521) is an international open access single-blind review journal published online.

Publication frequency: Semiyearly (1. January - June; 2. July - December).

Publication Fees: No fee required (no article submission charges or processing charges).

Digital Object Identifier DOI: [10.21533/pen](https://doi.org/10.21533/pen)

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1. January - June;
2. July - December.

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Helminth-Derived Product(S): Source for Potential Therapeutic

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Abstract

Helminth parasites that inhabit mammalian body surfaces have a highly evolved relationship with the immune system. Many of these resident helminths carry out functions to ensure their survival in the hosts. To attain this objective helminth parasites adopt immunoregulatory mechanisms to counter host's hostile immune response. Indeed, immunomodulatory molecules have been discovered in the worm's extracts and in their excretion/secretion. In this review, we discuss the state of our understanding of the interplay between helminths and immune pathways. We also highlight the key challenges that must be confronted in identification of the helminth-derived molecules involved in immune modulation. We consider whether helminth-derived signaling hold promise for the design of novel therapeutic approaches for the treatment of inflammatory disorders (inflammatory bowel disease, allergies, and autoimmune diseases).

1. INTRODUCTION

Parasites and bacteria produce a wide range of molecules that can modulate eukaryotic immune responses. These include the bacterial enterotoxins, parasite-derived excretory-secretory products. These molecules can subvert protective mechanisms in order to facilitate pathogen colonization and persistence. In many instances they are able to inhibit inflammatory responses. In fact, there are examples of immune modulators that can exert either stimulatory or suppressive effects depending on the molecular nature, mode of delivery, timing and dose of exposure to the agents as well as characterization of the modes of action so that synthetic analogues that mimic the effects can be generated. There is presently great interest in the therapeutic exploitation of these factors, for example as a means to stimulate enhanced immune responses to a new generation of subunit vaccines or to inhibit deleterious immune mediated diseases [1, 2].

Exposure to commensal and pathogenic microorganisms

strongly influences our immune system. Exposure to parasites and bacteria was frequent before humans constructed their current highly hygienic environment. Today, in highly industrialized countries, the contact between humans and the microorganism is dramatically diminishing. Congruent with the decline in helminthic and bacterial infections is an increase in the prevalence of autoimmune and inflammatory diseases. Incidence of some of these autoimmune and inflammatory diseases is now on the rise in the developing countries that are making rapid economic growth as people are adapting to more hygienic life-style due to affluence. It is possible that exclusion of many of the parasites and bacteria from the environment has permitted the emergence of immune-mediated diseases [3]. It is believed that parasites and humans have been co-evolved in the evolutionary history and the maintenance of an immunoregulatory environment may be promoted by parasites to ensure their survival in the hosts. A breakdown of this regulation that involves multiple levels of host regulatory cells and cytokines, is likely to

result into pathological disease.

In this review we will highlight the process in which helminths modulate both the innate and the adaptive arms of the immune system. It is our view that identification of the microbes-derived molecules involved in immune modulation will pave the way to novel therapeutic approaches for the treatment of inflammatory diseases. Here, we provide an overview of the findings from animal models and clinical studies, and additionally explores the potential for translation of these findings to the clinic.

2. BACKGROUND

Intestinal helminth infections are of particular interest because they have strong immune regulatory effects on their host [4,5]. The most common helminth infections humans are caused by geohelminths (also known as intestinal and soil-transmitted helminths). Geohelminth parasites include *Ascaris lumbricoides*, *Trichuris trichiura* and hookworm (*Ancylostoma duodenale* and *Necator americanus*). Other important helminthic infections in humans include schistosomiasis, fascioliasis and filariasis, which are tissue-dwelling parasites.

Currently, there is much interest in whether helminth-associated immune regulation may ameliorate allergy and autoimmune diseases, with investigations in both laboratory models and human trials. Analyses have consistently revealed bioactive molecules in extracts of helminths or in their excretory/secretory products that modulate the immune response of the host. Here, we illustrate the range of immunomodulatory molecules in selected parasitic trematodes, cestodes and nematodes, their impact on the immune cells in the host and how the host may recognize these molecules. Several years ago, we demonstrated that treatment with the extracts of *Dipetalonema vitae* male worms (a filarial nematode parasite) resulted into immunosuppression thereby enhancing the parasitemia in infected animals [6]. Recently, a tegumental coat antigen was isolated from the cestode parasite *Fasciola hepatica* (FhTeg) that had shown suppressive effect in vivo by directly targeting dendritic cells, impairing their ability to drive Th1 responses. In addition, FhTeg inhibits the ability of mast cells to drive the Th1 immune response by suppressing cytokine secretion (TNF- α , IL-6, IFN- γ , and IL-10) and ICAM1 expression in mast cells stimulated with LPS or heat-inactivated Bordetella pertussis Ag [7].

Symbiotic bacteria may be present in some nematode parasites. Lipopolysaccharide-like molecules have been detected in sterile products of filarial nematode *Onchocerca volvulus* stages, which could originate from Wolbachia bacteria related to Gram-negative Rickettsiales. Wolbachia bacteria are known to be abundant in the hypodermis and the female reproductive organs of *O. volvulus*. It has been demonstrated that

monocyte/macrophage would be a major target cell for immunomodulatory parasite-derived and intraparasitic, bacteria-derived molecules, thereby contributing to the host's cellular hyporesponsiveness [8].

Treating animals with helminths (eggs, larvae, extracts) causes dampening, and in some cases prevention of allergic and autoimmune diseases. Clinical studies showed that administration of eggs of *Trichuris suis* (a parasitic nematode of swine) reduces the disease severity to patients with inflammatory bowel disease [9]. Trials using colonization of helminths like *Necator americanus* (hookworm) or *T. suis* (porcine whipworm) show that they are safe and may be effective therapies for the control of the aberrant intestinal inflammation seen in Crohn's disease and ulcerative colitis [10]. Treatment with *Schistosoma mansoni* and *Ancylostoma caninum* soluble proteins significantly suppressed the inflammation and ameliorated trinitrobenzene sulphate (TNSB)-induced colitis [11]. Similar inhibitory effect on intestinal inflammation was observed when TNSB-induced mice received larvae of *Heligmosomoides polygyrus*, a duodenal nematode parasite [12].

Of note, *T. suis* when used in a randomized, double-blind, placebo-controlled clinical trial in Denmark against allergic rhinitis, no therapeutic effect was seen [13]. This raises the question whether the effectiveness of helminth therapy, especially with live intestinal nematodes will be disease specific, and may not be efficient enough against all inflammatory diseases. Further, such variation may be related to host's genetic or to environmental factors including the composition of bacterial population that colonizes the host's gut.

Multiple sclerosis (MS) is an inflammatory autoimmune demyelinating disease affecting the Central Nervous System (CNS), in which Th1 and Th17 cells appear to recognize and react against certain myelin sheath components [14,15]. Mucosal tolerance has been considered a potentially important pathway for the treatment of autoimmune disease, including MS and experimental conditions such as experimental autoimmune encephalomyelitis (EAE). It has been demonstrated that treatment with soluble products from the nematodes *T. suis* and *Trichinella spiralis* induces significant suppression of symptoms in EAE, a validated animal model for multiple sclerosis [16]. This amelioration in EAE pathogenesis is associated with the inhibition of TNF- α and IL-12 secretion by TLR-activated human DCs. Furthermore, helminth-primed human DCs differentially suppress the development of Th1 and/or Th17 cells [17].

The incidence of type 1 diabetes (T1D) is increasing dramatically in the developed world. While there may be several reasons for this, improved hygienic conditions and public health measures have impacted our

interactions with certain infectious microorganisms, especially with helminths. Therapeutic potential of both live infections as well as helminth-derived products has been explored in the animal models of T1D [18]. It was shown that infection with *Schistosoma mansoni* could prevent diabetes onset in NOD mice [19]. Other studies confirmed the potent effect of other helminth infections on T1D in NOD mice including *Trichinella spiralis*, *Heligmosomoides polygyrus* [20,21]. Protection was associated with an augmented Th2 response, increased generation of regulatory T (Treg) cells, production of IL-10 by T and B cells and reduction in infiltrating CD8⁺T cells in the pancreas; all of which would have the potential to inhibit diabetes onset. Interestingly, *H. polygyrus* infection induces non-regulatory T cells that produce IL-10 independently of STAT6 signaling. This signifies deficiency of Th2 response, and in this Th2-deficient environment IL-10 is essential for T1D inhibition [21]. The use of defined parasite products to influence diabetes onset will provide an additional way of addressing mechanisms of diabetes prevention.

A number of studies have now investigated the immunomodulatory mechanisms of helminth-derived soluble molecules in controlling allergy. [3,22]. As earlier as 1983, we tested the hypothesis whether Filarial nematode *D. viteae* could produce substances that might down-regulate hosts' allergic responses [23]. In this report, we described that a dialyzable fraction of *D. viteae* male worms' released product inhibited passive cutaneous anaphylaxis (PCA, anti-ovalbumin and ovalbumin). In subsequent studies other researchers described the immunomodulatory activity of ES-62, one of the excretory/secretory products of the filarial worm, *Acanthocheilonema viteae* (some scientists prefer to term *Dipetalonema viteae* as *Acanthocheilonema viteae*). Molecular characterization has shown that ES-62 is a 62-kDa phosphorylcholine (PC)-containing glycoprotein. ES-62-treated mice were protected in a cutaneous model of immediate-type hypersensitivity induced by oxazolone [24]. This ES-62 product has been shown to interact with a variety of cells of the immune system including B and T lymphocytes, dendritic cells, macrophages and mast cells. It appears that that ES-62 acts on TH17 responses rather than TH1 responses [25]. It has been shown that mice infected with nematode parasite, *H. polygyrus* are protected from allergic airway inflammation. Further, when the excretory-secretory material from this parasite was added to sensitizing doses of ovalbumin, the subsequent allergic airway response was suppressed, and this condition was associated with a lower ratio of effector [CD4 (+) CD25 (+) Foxp3 (-)] to regulatory [CD4 (+) Foxp3 (+)] T (Treg) cells, and reduced Th1, Th2 and Th17 cytokine production [26]. Of note, allergic asthma is less prevalent in countries with parasitic helminth infections, it appears that helminth-derived molecules can divert the immune system towards an anti-

inflammatory phenotype and may have therapeutic potential in inflammatory diseases.

3. MECHANISMS OF ACTION

Evidence from human studies and mouse models shows that infection with parasitic helminths has a suppressive effect on the pathogenesis of some inflammatory diseases [1, 5]. The overall paradigm in the way the helminth-derived products mediate immunomodulation appears to demonstrate their ability to impact the various immune activation pathways leading to a reduction in inflammation. Some helminthes and their products inhibit inflammatory responses through effects on both the innate and adaptive immune response (Fig.1). Helminth suppression of immunopathology generally involves CD4⁺ regulatory T cells (Tregs, either Foxp3⁺ or Foxp3⁻), IL-4-responsive cells, and TGF- β , IL-10 and Th2 cytokines. The potential regulatory role of CD8⁺ Tregs, regulatory B cells has not yet been clearly defined.

The favored mechanism is that microbial and parasite-derived products interact directly with pathogen recognition receptors to subvert proinflammatory signaling via T regulatory cells (Tregs), thereby inducing anti-inflammatory effects and control of autoimmune disease. An important immunoregulatory cytokine that participate in inducing tolerance is IL-10. This cytokine is produced by Foxp3 positive Treg cells (e.g. Tr1) [27, 28]. Although this may suggest that Tr1 cells are developmentally related to Treg cells, an alternative possibility is that IL-10 gene induction does not require Foxp3 expression [29]. One of the important components in immune activation implicates the role of TH17 subtype helper cells that secrete IL-17 cytokine [30]. Co-expression of T-bet and ROR γ t, which is found in subsets of IL-17-producing T-helper cells, may be an evolutionarily conserved transcriptional program that originally developed as part of the innate defense against infections but that also confers an increased risk of immune-mediated pathology [31]. The relationship between the various effector (Th1, Th17) and regulatory (Treg, Tr1) T cell subsets still needs to be clarified, in terms of antigen-specificity, development, and function. It seems that helminth-derive substance(s) may have impact on the innate immunity by targeting dendritic cells (DCs) and by regulating TLR signaling pathway. They may also down-regulate the Th1 response and polarize hosts' response towards Th2 response (Fig.1). By balancing these responses, helminths product(s) can reduce inflammation in autoimmunity and in other inflammatory disorders.

One approach would be to identify and isolate helminth-derived molecules that will facilitate iTreg cell induction. Another possibility would be ex vivo expansion and reinfusion of Treg cells, a strategy being attempted for

the treatment of autoimmune diabetes [32, 33]. Specifically expanding Treg cells might facilitate Treg cell localization to the gut and enhance suppressive activity. Thus, understanding the mechanisms of immune homeostasis to the intestine may provide insight into the pathogenesis and treatment of inflammatory bowel disease, colitis, and diabetes type 1. This may also apply to inhibiting Multiple Sclerosis pathogenesis as it has previously been shown that suppression of IL-17 and enhancement of Treg response are involved in amelioration of EAE pathogenesis [14,15]. As regards to inhibiting allergy/asthma, it is likely that helminth-derived molecules will inhibit mast cell activation induced by high affinity IgE receptor FcεRI, a critical receptor in patients with IgE-mediated allergy/asthma.

4. CONCLUSION AND FUTURE DIRECTION

Helminthic worms have evolved strategies to manipulate the host immune system, some of which may lead to a reduction in inflammation. Characterization of the ways in which these organisms mediate an anti-inflammatory response and identification of parasite-derived molecules involved in immune modulation will contribute to the discovery of novel therapeutic approaches for the treatment of inflammatory disease.

Although helminth infections are implicated in protecting the host against immune-mediated diseases, they are also believed to cause significant morbidity and in some cases mortality in endemic areas through their effects on nutrition, growth and cognition; and they have deleterious effects on vaccine immune responses. Thus, it might be more practical to focus on individual helminth-derived immunomodulatory molecules to selectively induce regulatory immune responses (particularly those expressed during the chronic phase of infection) and avoid any possible side effects of natural worm infections. The introduction of live helminths in intestine may favor the outgrowth or suppression of certain bacteria in the gut, which in turn can impact host's immunity. Hence, the priority would be to focus on the molecules released by helminth parasites (as excretory-secretory products). Clearly, further progress needs to be made in identifying parasite derived molecules, the ways in which they interact with the immune system and how they mediate immunomodulation in the genetically heterogeneous human population.

Much remains to be resolved regarding identification of potential helminth-derived biomodulators, timing and dose of exposure to the agents as well as characterization of the modes of action so that synthetic analogues that mimic the effects can be generated. Analyses have consistently revealed bioactive molecules in extracts of helminths or in their excretory/secretory products that modulate the immune response of the host. It is our view

that parasitic helminths are an untapped source of immunomodulatory substances that, in pure form, could become new drugs (or models for drug design) to treat disease. There are many examples of the partial characterization of helminth-derived immunomodulatory molecules, but these have not yet translated into new drugs, reflecting the difficulty of isolating and fully characterizing proteins, glycoproteins and lipid-based molecules from small amounts of parasite material. However, this should not deter the investigator, since analytical techniques are now being used to accrue considerable structural information on parasite-derived molecules, even when only minute quantities of tissue are available. With the introduction of methodologies to purify and structurally-characterize molecules from small amounts of tissue and the application of high throughput immunological assays, one would predict that an assessment of parasitic helminths will yield a variety of novel drug candidates in the coming years.

In order to develop non-living parasite-derived therapeutic agents, the molecular motifs that are responsible for protection from immunopathology must first be identified, their cellular targets need to be characterized and gene families implicated in immunomodulation have to be defined. A few molecules have already been identified as potential candidates for immunotherapy. It is important that efforts continue to bring these molecules to the clinical setting as they hold potential for human therapy of immunopathological conditions.

APPENDICES

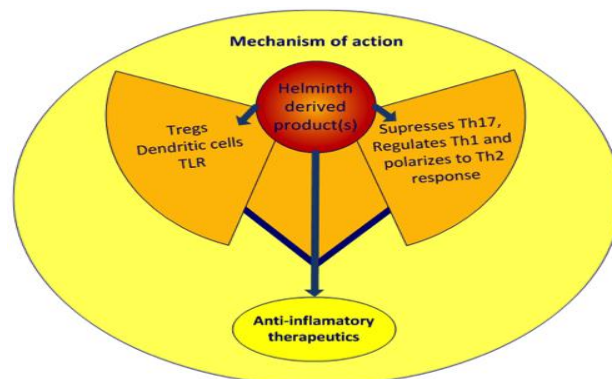


Figure 1 - Helminth-derived product(s) are potential candidates for immunotherapy. These molecules mediate suppression through CD4⁺ regulatory T cells (Tregs, either Foxp3⁺ or Foxp3⁻), modulatory dendritic cells (DCs), and by impacting TLR signaling. Regulatory responses induced by helminth-derived substances include suppression of Th17, Th1 response and polarization to Th2 response. Engagement of TLR on DCs may determine the Th2 polarization of the T cells.

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A Survey of Aircraft Materials: Design for Airworthiness and Sustainability

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Abstract

New developments in material science and its technologies find their best implementation areas in aircraft and space vehicles. Since the beginning of the powered flight, weight of airframes and systems are needed to be reduced. They are developed and built by light, durable and affordable materials through highly disciplined design, development, test and certification as well as manufacturing processes. Besides airframes, engineers are challenged to develop more efficient engines; both by reducing their weights and improving their aero-thermodynamic properties, sustaining higher operational and safety reliabilities along with complying stringent emission and noise restrictions. These conditions are increasing the demand for the development and the utilization of advanced lighter, stronger and durable materials and alloys, ceramic coatings and relevant manufacturing processes.

In this study, current trends and future expectations from material technologies in general; for accomplishing higher expectations for future lighter airframes, aircraft systems and engines, are reviewed.

Keywords: Advanced Materials, Aerospace Structures, Airworthiness Regulations, Concurrent Engineering and Surrogate Models.

1. INTRODUCTION

Civil Transport Aircraft (CTA) design, development, test, certification, production and related system technologies have been developed in parallel with the development levels of countries owning these technologies and industries. A typical CTA, shown as a generic design in Fig. 1, depends on several technological areas and uses various end products of these technologies as shown in Fig. 2. Production of a CTA, its roll out from the final assembly line is the end product of a highly diversified supply chain of; systems, major assemblies, components, parts and equipment depicted as a pyramid in Fig. 3.

Civil transport aircraft are first needed to be designed to fully comply with airworthiness requirements. In this perspective, present and future developments in materials are evaluated by Aircraft Structure Engineers; Designing for Structural Integrity, Fatigue and Damage Tolerance. Even at the Pre-Feasibility Phase of an aircraft project,

Structure Engineers must develop master Design Documents defining several objectives, approaches and standards starting with its basic criteria for which the aircraft structure would be designed and certified to ensure compliance with airworthiness requirement, including durability, inspection interval and threshold, frequent buckling and reparability of the structure. Allowable stresses are generated by analysis supported by rigorous test evidences by the design teams. For the durability criterion, the structure must be designed to demonstrate sufficiently high fatigue endurance throughout its Design Service Goal (DSG) to achieve two durability of the structure and minimize the number of areas prone to fatigue damage.

The development and implementation of new materials and manufacturing processes for aerospace application is often hindered by the high cost and long time span associated with current qualification procedures [1]. The

data requirements necessary for material and process qualification are extensive and often require millions of dollars and multiple years to complete. Furthermore, these qualification data can become obsolete for even minor changes to the processing route. This burden is a serious impediment to the pursuit of revolutionary new materials and more affordable processing methods for air vehicle structures. The application of integrated computational materials engineering methods to this problem can help to reduce the barriers to rapid insertion of new materials and processes. By establishing predictive capability for the development of microstructural features in relation to processing and relating this to critical property characteristics, a streamlined approach to qualification is possible [1].

Bringing a CTA Program to technical and commercial successes is not a straight forward journey and world civil aviation history may have more failure stories than the success stories. Availability of advanced materials is essential but utilizing them successfully and affordably is another further technological challenge. Integrated Product and Process Design (IPPD) and Concurrent Engineering (CE) disciplines are widely developed and are being implemented in this perspective especially in the last twenty years [2]. IPPD and CE disciplines utilizes today's advanced Product Life Cycle Management (PLM) tools, software, process, etc; a knowledge based environment help engineers to turn ideas and concepts to certified and commercially successful CTAs in to the market. Product Life Cycle Management (PLM), depicted in in Fig. 4 (*authors are thankful to Mr. Mustafa Ceren, Informatik, Turkey, for providing the figure*), provides engineering design, analysis, documentation and the integration of the overall product information environment with tools also named as Product Development System (PDM). All of these engineering disciplines and tools do not automatically guarantee low weight and durable airframe developments. Examples for successful designs were achieved in aeronautical and space vehicles, but structural designs which are ended with catastrophic failures or overdesign structures caused transportation of excess empty weights instead of revenue generating payloads, throughout the service life of the aircraft, have become real life experiences in aviation.

Fig. 5 shows the risk matrix of seven major risk areas of a CTA design to production program and these risks are related with the phases of the program, whereas if there would be a major problem related with the corresponding risk, it is too late to cure it at that stage of the program. Right Model means that the conceptual design of a new aircraft should be better or superior than its nearest competitors both technically and commercially just from the beginning of the program and must fill a Niche Market which is clearly visible in world CTA operation environment.

First challenge to reach to the "Right Model" during Concept Design of a new CTA; the Empty Weight versus Maximum Take of Weight ratio, which is also referred as Structural Efficiency of an aircraft is needed to be minimized within the design performance goals and airworthiness compliance constraints. Fig. 5 also gives examples for the weight ratios of a group of well-known CTAs [3].

Structural Efficiency is directly related with the fuel consumption; cost of fuel plus the cost of released carbon-dioxide emission. On the other hand reducing the empty weight is inversely effect structural strength of the airframe and the aircraft must also comply with the very stringent structural strength and service life durability requirements as per civil aviation regulations. These two conflicting two challenges make the material selection, design approaches and the manufacturing techniques quite important for the airframe of a new CTA.

It is envisioned by the world civil transport aviation sector that energy efficiency and overall productivity of next generation CTAs must be improved between 60~70% by 2030~2040 as illustrated in Fig. 6 [4].

In aviation this can be made possible primarily in two ways; by reducing the overall fuel burn and by increasing the engine efficiency. Commercial aircraft are expected to be dramatically leaner, cleaner and quieter in the next 25 years, but manufacturers will have to decide how far they want to push technology, and airlines must decide how much they are willing to pay for efficiency.

NASA initiated Next Generation Aircraft Concept Design studies [4] for aircraft entering into service by 2030 - 2035 as 70% reduction in fuel burn and 70% reduction in emissions release are targeted. In these studies, the contribution of the structure to the takeoff weight of the aircraft is aimed to be reduced 5%, whereas the total empty weight is aimed to be reduced about 30%. Moreover, propulsion system's contribution to the takeoff weight is aimed to be reduced by 3%. Besides, new propulsion systems' specific fuel consumption must be reduced down to 14 g / [kN s]. In order to achieve these goals, new materials, new processes, utilization of integrated product and process design methods will be needed for next generation aircraft [5], [6]. NASA N+3 2030-2035 subsonic aircraft concepts are shown in Fig. 7.

2. MATERIALS: AVAILABLE AND IN DEVELOPMENT

Two completely different trends are competing against one another within the aviation industry this decade [7]. One way or the other, the issues are just too complex and the existing technologies, resources and know-how just too deeply entrenched. There has been an ongoing contest between composites and metal construction in aircraft. In

a quest for distance, fuel economy, quietness, and cabin environment aircraft makers have invested billions in the use of composite materials in place of aluminum. Commercial applications, Boeing with the Dreamliner 787 that has set the standard for moving composites from use on the boundary to using them as the primary structure.

The process has initially started in military aircraft design; in 1987 the F-15 was constructed from 49% aluminum, 32% titanium and just 2% composites, by 2005 when the F22 entered service it was made from just 16% aluminum, 39% titanium and 24% composites. For commercial aircraft composites, although carrying a higher material and construction cost, it is expected to result in lower life cycle operating costs. Composites natural partner is titanium providing key structurally stressed component strength and with the rise in composite use has been a corresponding rise in titanium use. Titanium use has been on a rise.

For narrow body aircraft such as the rapidly recovering short haul and commuter jet market for 100–200 seats, metal airframes are offering considerable benefits, particularly in terms of lower development risks and lower material costs. To counter composites claims of weight reduction and with it lower operating, particularly fuel costs aluminum producers have introduced new alloys which exhibit higher strength properties allowing thinner gauges to be used and saving on weight. For example has brought out a new aluminum lithium alloy is used in fuselage skins, stringers, frames and floor beams. The alloy has been specified by Bombardier on its new C Series aircraft aimed at the 100-149 seat market [7].

Direct comparison of material properties between aluminum alloys and composites is not possible. The design drivers for the materials are significantly different, and therefore a comparison can only be made on a case-by-case basis. Detailed analyses need to be performed to determine which material is best suited for a specific structure. Scientific studies, material qualification and design implementation of new materials are continuing in aluminum and titanium alloys, composite material processing, manufacturing tools, monitoring and maintenance approaches are being continuously developed.

2.1. Chronology of Aluminum Development

The history of world aviation is closely related to aluminum and the history of creating aluminum alloys, and the more durable and reliable aluminum became, the higher, farther and safer airplanes flew [8]. The 2017-T4 Al-Cu-Mg-Mn alloy “Duralumin” was developed in Germany in the early 1900’s has been insensitively used in aviation starting with the first all-aluminum airplane, Junkers F13 manufactured in Germany in 1920. The

importance of corrosion was subsequently demonstrated by the development of Alclad 2024-T3.

After the World War II, the higher strength 7178-T6 was developed and was used on the first commercially successful jetliner, Boeing 707. Material selection progressed with the desire for higher fracture toughness; Alcoa developed 7475 to fill this need. It was first flown on the Panavia Tornado, and was selected for F16. A big technical and commercial success came with the development of the T77 temper for alloy 7150. For the first time, corrosion resistance was accomplished without having to sacrifice strength. Several materials now in the early stages of development include Al-Mg-Sc alloys with better corrosion resistance, lower density, and good welding characteristics. New generation Al-Li alloy 2097 with high resistance to fatigue crack growth is being developed for the bulkheads of high performance aircraft.

Recent alloy developments have produced a new generation of Al-Li alloys which provide not only density weight savings, but also many property benefits such as excellent corrosion resistance, good spectrum fatigue crack growth performance, a good strength and toughness combination and compatibility with standard manufacturing techniques. This results in well-balanced, light-weight aluminum alloys [9]. Finally, Al-Li alloys provide many property benefits over previous Al alloys and are often competitive with the performance composites can offer for many aerospace applications. Chronology of the development Aluminum alloys and latest Al-Lithium alloys development by Alcoa Company is shown in Fig. 8.

2.2. Current Usage of Composite Materials for Airframe Structures

The use of Fiber Reinforced Composite Materials have been continuously increasing since 1990s as shown in Fig. 9 and new Boeing 787 Dreamliner and Airbus 350 XWB series aircraft are utilizing highest amount of composite materials [10].

The A350 XWB’s airframe materials were selected for their optimum qualities in uses throughout the jetliner – from composites in the fuselage, wings and tail, to advanced metallic in such major components as the landing gear, engine pylon and structural beams. Referred as intelligent airframe, Airbus’ philosophy was about using the best material for each individual application. Airbus’ extensive application of composites – comprising 53 per cent of the overall airframe (compared to 11 per cent in the A330) – benefits from the design and manufacturing advances for such lightweight, strong and durable materials. Their advantages on the A350 XWB begin with reduced development times and higher production rates on the final assembly line; while contributing to lower overall aircraft weight, along with

proven in-service durability, reduced corrosion and fatigue, as well as lower maintenance costs.

Maintenance will be streamlined with Airbus' focus on improved and simplified aircraft systems for the A350 XWB – including hydraulics, electronics and power generation – which also enhance the aircraft's improved operating economics. The use of composites in the fuselage, wing and tail assumed to reduce maintenance tasks by creating a more "intelligent" airframe with increased resistance to corrosion and fatigue during the jetliner's lifetime.

Advanced metallic materials also have found their place on the A350 XWB, including low density/high performance aluminum-lithium alloys that provide increased stiffness and resistance at lower weight in floor beams, frames, ribs and landing gear bays. The latest titanium alloys are applied in main landing gear supports, engine pylons, and attachments.

The A350 XWB's major fuselage sections are created by the assembly of four large panels each, which are joined with longitudinal riveted joints. The 4-panel concept also is aimed to provide considerable weight savings, as the use of longer panels requires fewer circumferential joints and relies more on lighter longitudinal joints. This weight savings also results from better optimization of each panel for its application. The use of fewer, longer sections also means fewer joints overall – which are placed for load and weight optimization.

Another benefit is better reparability in operational service, as an individual panel can be replaced in the event of significant damage – avoiding major repair work that could require extensive composite patching. Composite material usage in A350XWB aircraft is shown in Fig. 10 [11].

Responding to the overwhelming preference of airlines around the world, Boeing Commercial Airplanes in 2004 launched the 787 Dreamliner, an all-new, superefficient airplane. An international team of top aerospace companies builds the airplane, led by Boeing at its Everett, Wash., facility near Seattle and in North Charleston, S.C. In addition to bringing big-jet ranges to midsize airplanes, the 787 provides airlines with unmatched fuel efficiency, resulting in exceptional environmental performance. The airplane uses 20 percent less fuel than today's similarly sized airplanes. The 787 also travels at a similar speed as today's fastest twin-aisle airplanes, Mach 0.85. Airlines also realize more cargo revenue capacity -- a 20 to 45 percent advantage over today's similarly sized airplanes.

Passengers also enjoy improvements on the 787 Dreamliner, from an interior environment with higher humidity to more comfort and convenience. The key to the exceptional performance of the 787 Dreamliner is its

suite of new technologies and its revolutionary design. Composite materials make up 50 percent of the primary structure of the 787, including the fuselage and wing.

At the heart of the 787 design is a modern systems architecture that is simpler, more functional and more efficient. For example, onboard health-monitoring systems allow the airplane to self-monitor and report systems maintenance requirements to ground-based computer systems. Advances in engine technology are the biggest contributor to overall fuel efficiency improvements on the Dreamliner. The 787 features new engines from General Electric and Rolls-Royce companies that represent nearly a two-generation jump in technology.

The design and build process of the 787 has added further efficiency improvements. Boeing and its supplier partners have developed new technologies and processes to achieve efficiency gains. For example, manufacturing the 787 fuselage as one-piece sections has eliminated 1,500 aluminum sheets and 40,000 - 50,000 fasteners per section. More than 50 of the world's most capable top-tier supplier partners are working with Boeing to bring innovation and expertise to the 787 program. The suppliers have been involved since the early detailed design phase of the program and all are connected virtually at 135 sites around the world.

The 787 program opened its final assembly plant in Everett in May 2007 and in North Charleston in July 2011. First flight of the 787-8 Dreamliner occurred on December 15, 2009, followed by certification in August 2011. First delivery of the 787-8 took place on Sept. 25, 2011. Composite material usage in Boeing 787 aircraft is shown in Fig. 11 [12].

2.3. Titanium Alloys

Currently a greater amount of titanium is incorporated in to aircraft. This is connected with the fact that the share of the composite materials with which aluminum intensively interacts and corrodes in the new airplanes is being increased. Titanium is not subjected to these processes and results in increasing the life of components. Applications run from massive highly stressed, forged wing structures, and landing gear components, to small critical fasteners, springs and hydraulic tubing.

Titanium usage on Boeing aircraft has increased from 2% empty weight on the 737 to 17% on the 787. Titanium alloys now replace nickel and steel alloys in nacelles and landing gear components in newer airframes such as the Boeing 777, 787 and Airbus 380. Super-plastic forming/diffusion bonding has helped to increase the use of titanium alloys (Fig. 12) in new airframe designs, by lowering the cost through less machining, reworking and fewer component parts [13].

Ultra fine-grained titanium is characterised by exceptional mechanical properties, among which *high ultimate strength* and *high yield strength* are of utmost importance (Fig. 12). Classical coarse-grained titanium the relation (strength/density) varies around 70 to 120 (N·m/g) Alloy Ti6Al4V it varies around 200 (N·m/g). Ultra-grained titanium it is possible to predict the values (strength/density) = 270 (N·m/g) (Fig. 13).

2.4. Metal Bonding / Fiber Metal Laminates (FML)

The combination of metallic materials with fiber reinforced polymers into aircraft structural materials is commonly denoted as hybrid concepts or technologies. These concepts have their origin in the addition of reinforcing fibers into the bond line of thin laminated aluminum sheets [14]. Well-known examples of the FML are Arall (Aramid Reinforced Aluminum Laminates) and Glare (GLASSREinforced aluminum), aramid/glass fibers embedded in the epoxy system with aluminum layers, respectively. Glare is currently applied as skin material on Airbus A380 fuselage and as leading edges of the tail planes of this aircraft [15], [16], [17] (Fig. 14). Main advantages of FMLs over monolithic aluminum alloys are the increased fatigue and corrosion resistance. Compared to fiber reinforced polymer composites, FMLs have higher bearing strength and impact resistance and they are easier to repair. They also provide weight reduction by 15-30%.

A similar recent material is termed as CentrAl. The new, CentrAl concept comprises a central layer of FML, sandwiched between one or more thick layers of high-quality aluminum. FMLs consist of alternating layers of uni-directional impregnated fiber lamina and thin metallic sheets adhesively bonded together. This technique of coupling the metal with fiber shows improvements over the properties of both aluminum alloys and composite materials individually [18] (Fig. 15).

Aluminum metal bonding airframe structures have been widely used in Fokker70 and Fokker100 aircraft; metal bonding and intelligent use of composites have resulted with light airframe. With the proven excellent in service life; 11 million flight hours and nearly 10 million flight cycles, the structural integrity and durability which guarantee crack-free-life for 45.000 cycles, economical repair life for 90.000 cycles and superior corrosion resistance, a stretched version F120 Next Generation with new engines and complete new flight deck is being proposed. As shown in Figure 16 the proposed F120NG can have better “Structural Efficiency” even compared with new designs which are utilizing higher composite material usage. (*Authors are thankful to Mr. Rudi den Hertog and Mr. Maarten van Eeghen, NG Aircraft Company, Netherland for the information and the figure provided*).

2.5. Composite Materials for Future Airframe Structures

The use of composite materials and new concepts for the manufacturing technologies for new composite structures are expected to be increased in next generation aircraft. As examples by the improvement of 3-D Woven Pi-Preform Joints, creation of large integrated composite structures and sub-structures through composite pi-joints will be possible. It also prevents the exploitation of orthotropic properties of carbon fiber and limits out of plane failure modes. Moreover, size limitations found on pre-prep systems are removed since it is assembled in dry conditions (Fig. 17) [5]. Another concept is the Affordable Large Integrated Structures. Advancements in alloy, composite, and composite joint technology allow design flexibility toward utilized structures. The introduction of the Affordable Large Integrated Structures eliminates structural discontinuities and fastened assemblies, increasing structural efficiency, providing reduction in part count and weight (Fig. 17) [5].

Advancements in composites find their place in airplane skin too as a new skin concept is introduced. New protective skin weighs less than half of the current composite coatings with increased damage tolerance by the help of energy absorbing foam. The conductive skin over the foam protects the composite structure from lightening and also provides electromagnetic interference and environmental protection as shown in Fig. 18 [19].

Carbon nano-tubes are hexagonally shaped arrangements of carbon atoms bonded into a tube shape, sometimes with a single wall — called single-wall carbon nano-tubes or SWCNT — or multiple walls — called multi-wall carbon nano-tubes MWCNT. Carbon nano-tubes have many remarkable properties which we are only just starting to exploit. First of all, carbon nano-tubes are extremely strong, probably one of the strongest materials that is even theoretically possible. Although nano-tubes are only about a nanometer wide, they can be very long in comparison to their width, a useful property for strength (Fig. 19). Carbon nano-tubes are hexagonally shaped arrangements of carbon atoms bonded into a tube shape, sometimes with a single wall - called single-wall carbon nano-tubes or SWCNT - or multiple walls - called multi-wall carbon nano-tubes MWCNT.

Although the longest nano-tubes that have been synthesized are only a few cm in length, Nanocomp Technologies Inc. have taken a step towards making carbon nano-tubes into nano-tube fibers kilometers long. The fibers have the strength of spider silk and more than three times its shock-absorbing toughness. These fibers are both tougher and stronger than steel. The fibers have twice the stiffness and strength and 20 times the toughness of the same weight and length of steel wire [20] (Fig. 20).

2.6. New Materials in Aircraft Engines

A major effort underway in this area is the Advanced High Temperature Engine Materials Technology development which focuses on providing revolutionary high-temperature composite materials: to 425°C for polymer-matrix composites (PMCs); to 1250°C for metal-matrix / inter-metallic-matrix composites (MMCs / IMCs); and to as high as 1650°C for ceramic-matrix composites (CMCs) (Fig. 21).

Based on the preliminary designs of next generation conceptual engines, however, material temperatures approaching 1650°C are anticipated for the turbine inlet, thus requiring extensive use of CMCs throughout the combustor, turbine, and exhaust nozzle. One benefit of using CMCs is that they allow higher operating temperatures and thus greater combustion efficiency leading to reduced fuel consumption. Thanks to the low density of CMCs, compared with current technology, the use of CMCs in the hot section of the engine along with IMCs in the compressor is resulting in a 50% reduction in engine weight.

Ceramic matrix composite turbine blades and turbine materials are attractive due to their high temperature tolerance. Without the need to cool the turbine blades, compressor bleed will no longer be required and higher temperatures can be achieved with the combustor.

Ceramic-matrix composites research is aimed at developing the basic and applied technologies needed to fabricate structurally reliable ceramic composites reinforced with long or continuous ceramic fibers (Fig. 22). Like monolithic ceramics, these fiber-reinforced ceramics (FRCs) have lower densities, better oxidation resistance, and potential to operate at significantly higher temperatures than super alloys. However, unlike monolithic ceramics, FRCs display metal-like deformation behavior, non-catastrophic failure, and strength properties that is insensitive to processing- and service-generated flaws [21].

The use of CMCs in gas turbines would permit higher turbine inlet temperatures, which would improve turbine efficiency. Because of the complex shape of stator vanes and turbine blades, the development was first focused on the combustion chamber. A combustor made of SiC/SiC with a special SiC fiber of enhanced high-temperature stability was successfully tested for 15,000 hours [22]. SiC oxidation was substantially reduced by the use of an oxidation protection coating consisting of several layers of oxides [23].

Polymer-matrix composites (PMCs) are the lightest of the three types of composite materials under study and recent applications of PMCs in aircraft propulsion systems, such as General Electric's F-404 engine, have resulted in substantial reductions in both engine weight and

manufacturing costs. To realize the full advantages of PMCs in aircraft-propulsion systems, however, new composite materials must be developed with enhanced thermal-oxidative stability permitting their use at temperatures to 425°C [24].

Lightweight Fan / Fan Cowl can be achieved by the use of design optimization. Shape memory alloy nozzles (variable geometry nozzles) utilize a shape memory alloy actuated hinge that is able to be varied and controlled which allows for optimization of engine for given power setting and target condition. Active compressor clearance control provides higher compressor efficiencies by minimizing the blade tip losses by maintaining tip clearances which takes the form of variable, flexible surface maintained by electromagnetic actuators (Fig. 23) [5].

3. DESIGN FOR AIRWORTHINESS IN AIRCRAFT STRUCTURES

3.1. Airworthiness Regulations for CTA

In civil aircraft world; design, development, production and operation, personnel training, maintenance (MRO), air traffic control and all related sub-activities are regulated and controlled by international rules and organizations. The top regulating organization is the International Civil Aviation Organization (ICAO) established under United Nations in 7th December 1944 with Chicago Convention. Under ICAO rules several National Civil Aviation Authorities were established such as: EASA in Europe (European Aviation Safety Agency), FAA (Federal Aviation Authority) in United States. Turkey operates and maintains aircraft according to Turkish Civil Aviation Authority Regulations (DGCA-SHGM) which are fully compliant with EASA and FAA regulations. The complete Life Cycle of an aircraft; from design manufacturing and operation must be certified by the authorized organization.

Aviation Products, their Utilization and Organizations Approval (Aircrafts, Engines) Top Down Regulation Hierarchy of EASA is shown in Fig. 24 [25], [26]. Top regulations for airworthiness of civil aircraft are defined by Annex 8 of the Chicago Convention and EASA CS25 and FAA FAR25 regulations define the design and certification requirements for CTA (Large Aircraft).

3.2. Certification of Product and Parts and Appliances

3.2.1. Product

Design Organization Approval (Subpart J)

Product Organization Approval (Subpart G)

3.2.2. Aircraft (Type Certification) Certification Basis for Large Aircraft (CS25)

3.2.3 Engine (Type Certification) Certification Basis (CS-E)

3.2.4. Propeller (Type Certification) Certification Basis (CS-P)

3.2.5. Change to Type Certifications:

Design Organization Approval (Subpart j)

Production Organization Approval (Subpart G)

STC (Supplemental Type Certification)

Major Changes/Minor Changes

3.2.6. Parts and Appliances

ETSO Parts: (European Technical Standard Orders)

3.3. Reliability

The concept of Continuing Airworthiness is closely related with Reliability of aircraft and its systems. Relatively few systems are designed to operate without maintenance of any kind. For most systems there are two types of maintenance, one or both of which may be applied. In preventive or scheduled maintenance, parts are replaced, lubricants changed, or adjustments made before failure occurs. The objective is to increase the reliability of the system over the long term by preventing the aging effects of wear, corrosion fatigue, and related phenomena. Whereas, corrective or unscheduled maintenance is performed after failure has occurred in order to return the system to service as soon as possible. Such maintenance is performed at unpredictable intervals because the time to any specific unit's failure cannot be established ahead of time.

In general structural design load and damage considerations of airworthiness requirements (CS25 and FAR25) define Limit and Ultimate Loads. The Limit Load is defined as being the maximum load per life which may only cause a detectable damage to be found and repaired through maintenance. Ultimate Load is the 1.5 times of the Limit Load and it is allowed to cause only an acceptable but non-detectable damage which is referred as the Allowable Damage Limit [10].

3.4. Design Criteria for Fatigue and Damage Tolerance

The basic Fatigue and Damage Tolerance (F&DT) criteria against which the aircraft structure is designed and certified, is to ensure compliance with the airworthiness requirement, include durability, inspection interval and threshold, frequent buckling and reparability of the structure. Allowable stresses S_{allow} , are generated by analysis supported by a series of test evidences. The allowable stresses are dependent on the design geometry; the material used as well as in some cases the loading pattern. During the detailed sizing of an aircraft structure, Reserve Factors or Margins of Safety are calculated as,

$$MS = \frac{S_{allow}}{S_{equivalent}} - 1$$

Where, $S_{equivalent}$ is the maximum stress with stress ratio R that produces the same damage to the fatigue spectrum at the specific location.

Regarding the durability criterion, the structure must be designed to demonstrate sufficiently high fatigue endurance throughout its Design Service Goal (DSG), to achieve the following objectives:

- Ensure durability of the structure throughout its operational life.
- Minimize the number of areas prone to fatigue damage and development of cracking in service.

Structural detailed analysis, geometrical sizing and tests must demonstrate that the calculated fatigue life of the structure N_F is higher or equal to the DSG multiplied by an appropriate Scatter Factor (SF).

$$N_F \geq DSG \times SF$$

The value of the SF depends on the stress (Stress Life) design data used in the analysis.

The **threshold** for initial inspection of the structure should be defined as a design objective. Design precautions will be taken for the following objectives:

- Ensure the minimum inspection threshold will be equal to the target value.
- Ensure that any damage will not reach its critical size before the first inspection occurs.

Analysis and tests must demonstrate that service life of the structure N_C is higher or equal to the design objective inspection threshold T , multiplied by an appropriate SF.

$$N_C \geq SF \times T$$

The evaluation of the inspection threshold, using initial flaw concept, must ensure that cracks will not propagate from the initial defects to the critical sizes within the inspection threshold interval. This approach is applicable to Single Load path structures and Multiple Load Path structure where it cannot be demonstrated that load path failure, partial failure, or crack arrest will be detected and repaired during normal maintenance.

The repeat inspection interval is the time between two successive directed inspections during which any damage must not propagate from the detectable size to the critical size. Design precautions will be taken for allowing objectives:

- Provide damage tolerance capability of the structure.

- Ensure that any damage will be detected before it becomes critical within the targeted inspection interval.
- Maintain airworthiness through scheduled inspections.

Repeat inspection interval is derived from period of time during which damage is detectable, and the residual strength remains above the required levels. Consequently, the structural assessment should include a calculation of the period of failure crack in the critical location of the structure to develop from the detectable size to the critical size under residual strength loads. An inspection interval is then established by applying an appropriate scatter factor to this crack growth period, in order to ensure that the crack will be detected before the residual strength of the structure is compromised.

The detectable crack size is assumed in determining the inspection interval should be consistent with the capabilities of the proposed inspection method.

The crack growth analysis and crack propagation test must demonstrate that the period n_{det} , during which the crack propagates from the detectable size to the critical size, is higher and or equal to the required repeat inspection interval I , multiplied by an appropriate SF .

$$n_{det} \geq SF \times I$$

Frequent buckling should be avoided because it has an impact on the fatigue lives of the skin and webs and/or its surrounding structure. The fatigue load spectrum should be analyzed to ensure that the buckling will not occur more than specified number during the operational life of the aircraft. The allowed numbers repeated buckling should be substantiated by test under fatigue damage to structure buckling within the fatigue load range.

Repair-ability is a characteristic of the design and related to the ability of structure to incorporate an acceptable repair with the minimum of structural modification following the occurrence of reasonable damage. Repair-ability should be considered in the geometric design and detailed sizing of the aircraft. Repair-ability is enhanced is accessibility, serviceability and standardization are maximized and corrosion requirements are minimized. In addition, the maximum use of interchangeable components becomes desirable which can facilitate rapid repair and replacement. Repair-ability concept, limit and ultimate load capability of an aircraft structure is illustrated in Fig. 25.

4. INTEGRATED PRODUCT AND PROCESS DESIGN (IPPD):

As introduced in Section 1; the current qualification procedures for the development and implementation of

new materials and manufacturing processes for aerospace application is very costly and long process, selection of right materials and designs for airframe components which complies very stringent airworthiness requirements, Structural Engineers often feel as surrounded with too many constraints. It is a very difficult challenge to minimize the empty weight by accomplishing too many conflicting objectives and highly bounding constrains. But on the other hand, structural engineers now have methodologies and tools which enable them to generate innovative solutions to these challenges.

Integrated Product and Process Design (IPPD), Concurrent Engineering (CE) and Product Life-Cycle Management (PLM) tools (Fig. 4) and methodologies are well developed for robust, integrated and optimized design solutions [1999]. As being one example, Aerospace Systems Design Laboratory (ASDL) at Georgia Institute of Technology, Atlanta, USA, has been continuously improving these IPPD methodologies as well as implementation of new available tools through various research and design activities [27].

An exceedingly large number of scientific and engineering fields are confronted with the need for computer simulations to study complex, real world phenomena or solve challenging design problems. However, due to the computational cost of these high fidelity simulations, the use of neural networks, kernel methods, and other surrogate modeling techniques have become indispensable. Surrogate models are compact and cheap to evaluate, and have proven very useful for tasks such as optimization, design space exploration, prototyping, and sensitivity analysis. Consequently, in many fields there is great interest in tools and techniques that facilitate the construction of such regression models, while minimizing the computational cost and maximizing model accuracy. Reference [28] presented a mature, flexible, and adaptive machine learning toolkit for regression modeling and active learning to tackle these issues.

As being an instructive study to implement IPPD and CE methodologies as well as to utilize PLM tools, a CTA floor beam structural design and analysis conducted by reference [29] will be presented as an example.

4.1. Problem Definition

3-D view of the floor beams in the fuselage and simplified beam section are shown in Figure 25. Major design variables are selected as section dimensions shown in Figure 26 along with the material type (Aluminum/Titanium) and manufacturing method (NC/Sheet Metal). The Overall Evaluation Criteria (OEC) constructed to represent the overall expectations from the design is formulated as:

$$OEC = \alpha (W/W_{BL}) + \beta (C/C_{BL}) + \gamma (\text{Shape Criteria})$$

Where α , β and γ are weighting parameters for weight, cost and geometric constraints respectively. W and C are weight and cost of the component and W_{BL} and C_{BL} are baseline values to normalize weight and cost respectively.

The ultimate goal is to determine the values of design variables that minimize OEC while satisfying static structural constraints. Structurally it is required to ensure that the beams do not fail under the loads which they will be exposed during their complete life cycle. The constraints are defined as:

$$\text{Min } (MS_i) > 0 \quad i = 1,2,3,4,5,6$$

Max Deflection > defined values

Where MS_i is margin of safety due to any of six primary static stress failure criteria selected as:

- Shear buckling
- Bending buckling
- Combined shear-bending buckling
- Shear stress
- Axial stress
- Crippling

Deflection constraint has been rewritten so as to make it “higher than zero” constraint like MS, and it has been assumed as seventh MS.

4.2. Implementation of DOE-RSM Approach

Having selected weight, cost, OEC, and minimum and norm of the margins of safety – including deflection constraint – as responses, Design of Experiment (DOE) and Response Surface Method (RSM) have been realized by using JMP, a statistic software by SAS Institute, NC, and a 128-experiment custom model has been constructed by JMP commercial software [30]. Engineering simulations and modeling are performed by CATIA, MSC PATRAN/NASTRAN and several in-house structural analyses codes (representing the PLM environment). In this preliminary study, a simplified parametric cost model has been used [31]. Formulation is written for particular design as;

$$C = W a \cdot b + W \cdot c / Q$$

where C is manufacturing cost including material acquisition cost, W is weight, a , b , and c are parameters dependent upon material type and manufacturing method, and Q is production quantity.

Response Surface Designs (RSD) [32] are based on the assumption that complex relationships between design variables often examined through the use of sophisticated

and time consuming codes, can be represented by a quadratic equation. This response is a function of the most important design variables and their interaction. In cases where no prior knowledge exists as to which variables are important, a Screening Test (ST) has to be performed. The ST is used to identify primary contributing factors among a set of design variables at two-level (minimum and maximum settings) during the DOE phase. The Response Surface Methodology (RSM) encompasses a set of techniques by which relationships between a set of independent variables and their dependent functions can be studied empirically. The “response” is the outcome of each individual experiment and the response values are then used to create surface equation fits based on the various independent parameters. The surface fit equations are selected as with the form:

$$Y = \alpha_0 + \sum_i \alpha_i X_i + \sum_{i < j} \alpha_{ij} X_i X_j$$

The coefficients of this equation are determined through a three-level DOE. Since an equation involving too many variables is impractical, the number of variables must be reduced to a manageable size. Sensitivities and responses to the design variables are given in Fig. 27.

Response surface for the objective OEC of the beam design in this example is the Surrogate Model of the beam structural and geometrical design. With today's advanced PLM environment (Fig. 4) and tools, engineers can utilize Parametric Design and Surrogate Models connected as several serial and parallel design sub-activities and can perform optimization procedures for iterating and selecting the Best Affordable Design Solutions [28].

5. DISCUSSIONS AND CONCLUSIONS

As being one of the major and initial challenges in the design and development a new CTA, assuring a desired Structural Efficiency is quite complicated engineering process. Top decision makers give strategic decisions step by step during Pre-Feasibility, Concept Exploration, Preliminary Design and Concept Definition phases of the new CTA Program. They should be provided with precise solutions and clear alternatives in the decision making processes.

Authors evaluate that difficulties and risks, which are addressed in Section 1, can be turned to new challenges and opportunities if correct approaches will be used. First of all, structural design is a team work and good results can be achievable by common and well shared intelligence. Structural design teams first must respect the vast experiences of senior engineers and on the other hand young engineers must be knowledgeable and competent in using advanced methodologies and tools in a fast and accurate way. Innovative thinking can give

results with disciplined and systematic design iterations but right modeling always depends on real life experiences.

As outlined in Section 2, development new and affordable materials for aircraft structures will lead structural engineers to design and manufacture lighter and durable airframes. Reversely, aircraft industry will be seeking and demanding for new materials and processes for new airframe design applications. Aircraft structures will continue to utilize metallic and composite materials with different forms of design and manufacturing processes for near decade. Airplanes are always multi-material and the use of aluminum in aerospace is projected to grow along with the usage of composite materials.

In section 3 it is emphasized that CTA airframe designs are strictly regulated to ensure the continued

airworthiness of the aircraft throughout its life cycle as long as it operates. Aircraft structural engineers must well understand certification aspects of the structural design even down to very detailed component and small parts level. On the other hand regulators must also ease the qualification processes for new materials, with a parallel utilization of new technologies and processes by which these new materials and processes will be developed.

Aviation is expected to grow 6% annually and aircraft replacements can be realized earlier than expected. There will be a continuous demand for good structural engineering. As briefly explained in Section 4 available and continuous developments in IPPD, CE, PLM and all other engineering design tools and methodologies will enable structural engineers to develop better solutions and intelligent designs.

APPENDICES



Figure 1 - A generic CTA, is the end product of a diversified materials equipment and industrial/technical knowhow supply chain.

TECHNOLOGIES AND DISCIPLINES RELATED WITH AIRCRAFT



Figure 2 - Commercial Transport Aircraft Design, Development, Certification, Production and Operation (Life Cycle) utilizes several technologies and disciplines which are mainly developed as spin-off technologies of aerospace industry itself in decades.

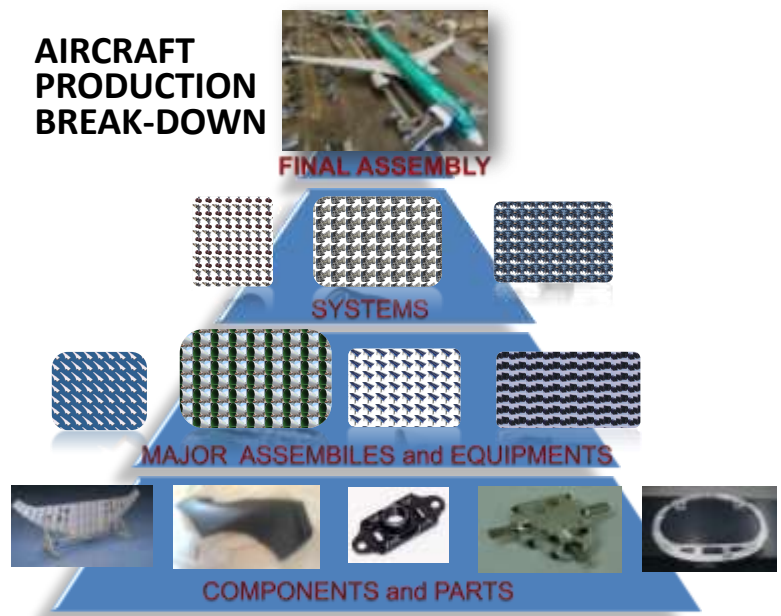


Figure 3 - The pyramid of the highly diversified supply chain of systems, major assemblies, components and parts of CTA production.

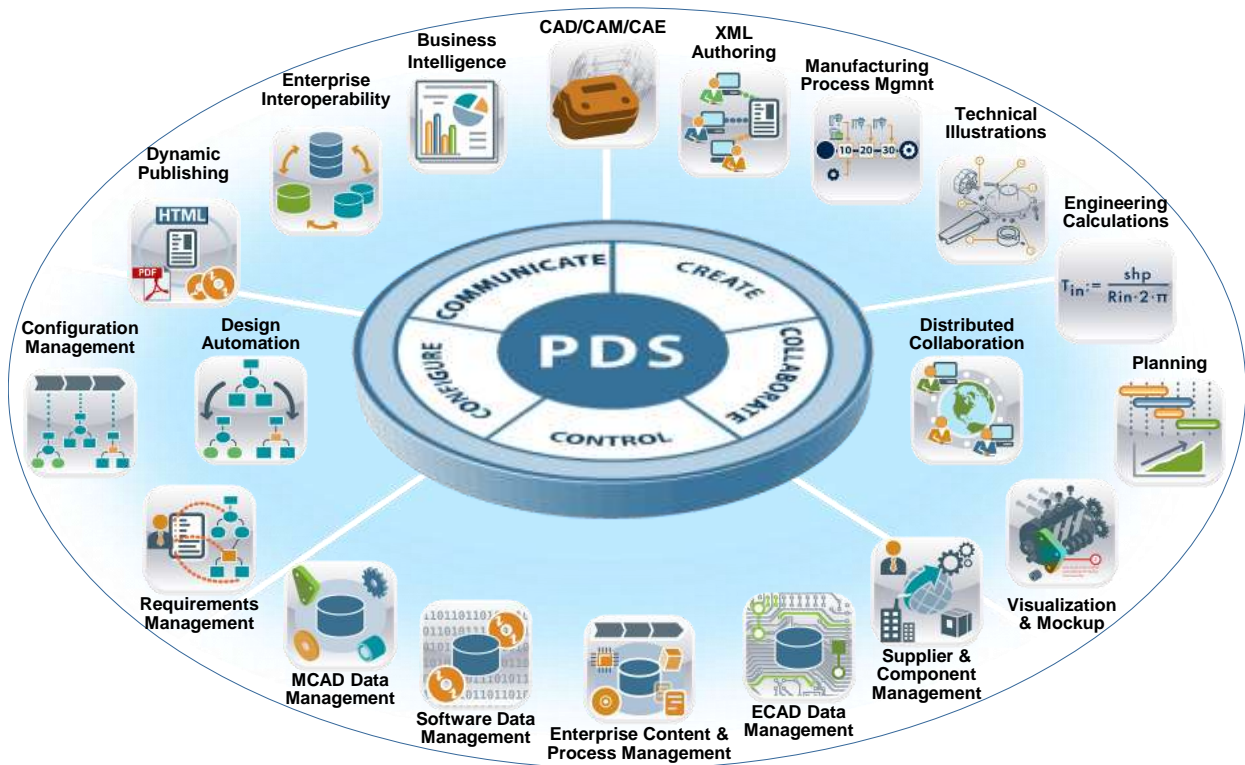


Figure 4 - Product Life Cycle Management (PLM) provides engineering design, analysis, documentation and the integration of the overall product information environment with tools also named as Product Development System (PDM) (Provided by Mr. Mustafa Ceren, Informatik, Turkey).

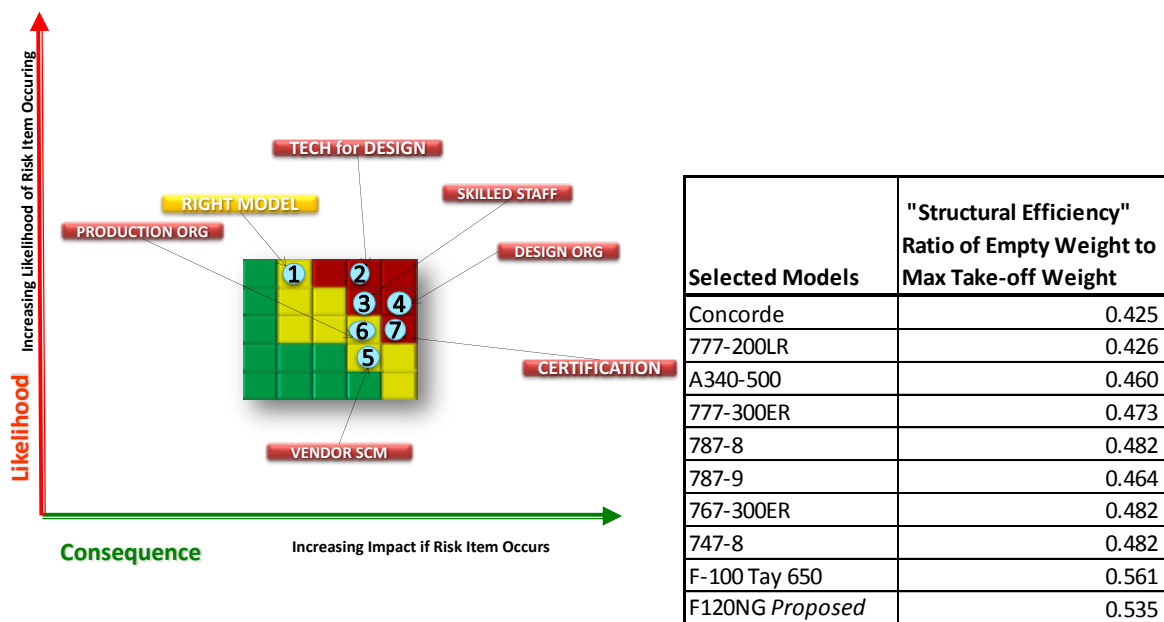


Figure 5 - CTA Design, Development, Certification and Production Program 7 Major Risk Areas for A New Indigenous Design and "Structural Efficiency" of the Selected CTAs [3].

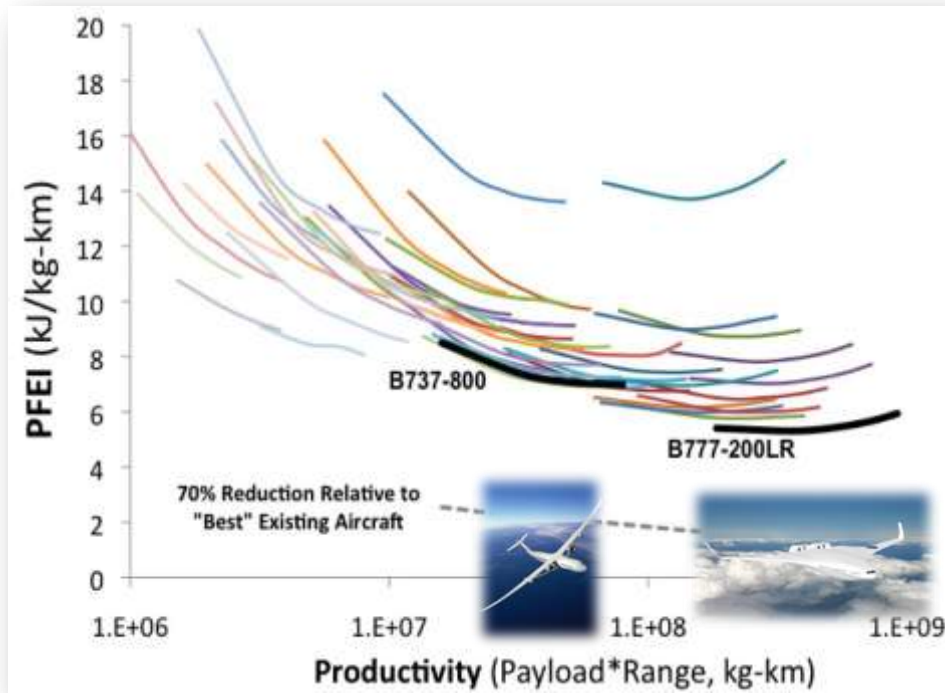


Figure 6 - Commercial Transport Aircraft Fuel Burn Goals for 2030-2035 70 % reduction in total fuel burn per seat-mile [5], [6].

NASA N+3 Future Subsonic Fixed Wing Aircraft

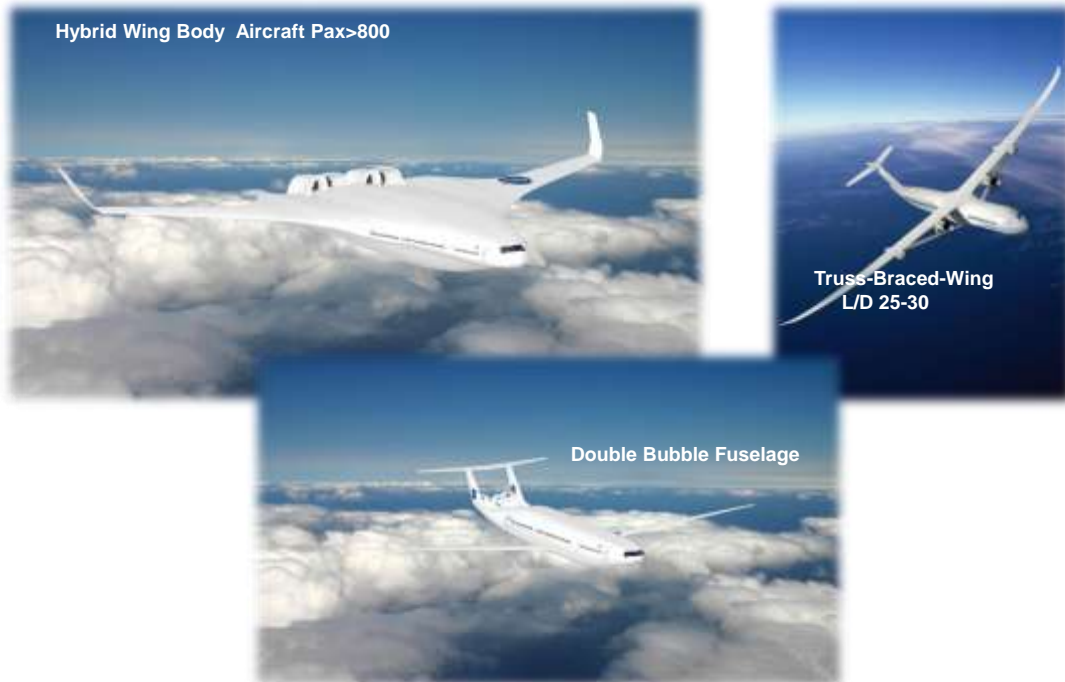
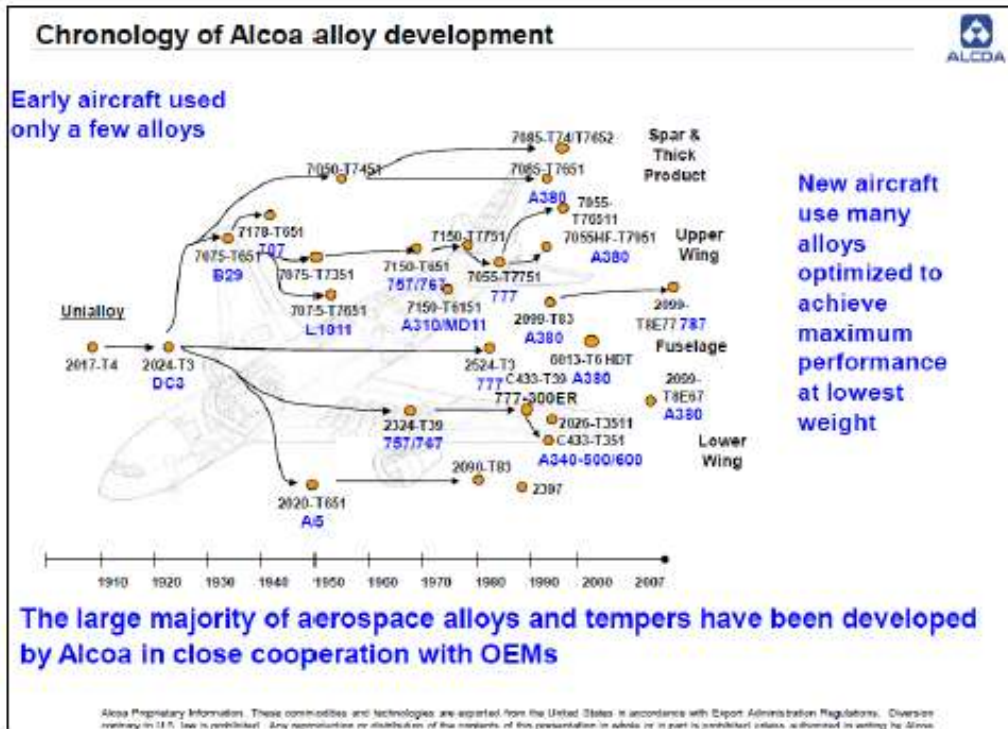


Figure 7 - Future Aircraft Concepts NASA N+3 for 2030-2035 [5], [6].



Alcoa: the Al-Li pioneer now rolls out a third generation of new corrosion resistant alloys for longer inspection intervals*

Exfoliation Corrosion: 3rd Gen Al-Li alloys are highly resistant to exfoliation corrosion

7150-T6511 specimens fastened and coated -- severe exfoliation propagated from coating defects after moderate seacoast exposure

Al-Li 2099-type specimens with no coating -- No exfoliation occurred after prolonged seacoast exposure

Stress Corrosion: 3rd Gen Al-Li alloys have doubled their resistance to stress corrosion

| Alloy Type | ST SCC Threshold Stress (KSI) |
|------------------------------|-------------------------------|
| 7150-T7751 (Conventional) | ~25 |
| 2024-T351 (Conventional) | ~10 |
| 2099-T86 (3rd Gen AL-Li) | ~50 |
| 2199-T8E80 (3rd Gen AL-Li) | ~45 |
| C14-type T8X (3rd Gen AL-Li) | ~45 |

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Figure 8 - Chronology of Aluminum Alloys Development and Aluminum Lithium Alloys (Copyright of Alcoa Company).

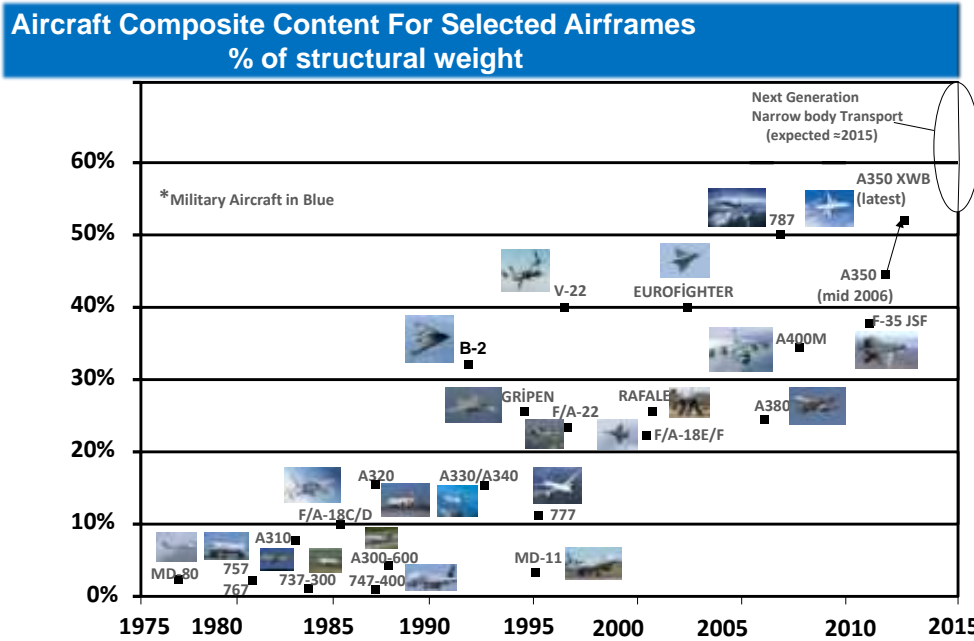


Figure 9 - Chronology of Composite Material Usage in Aircraft (Reproduced as based on [10]).



Figure 10 - Composite Material Usage in Aircraft Industry, Airbus A350 XWB Aircraft, Copyright of Airbus [11].



Figure 11 - Composite Material Usage in BOEING 787 Aircraft, Copyright of Boeing Commercial Airplanes, [12].

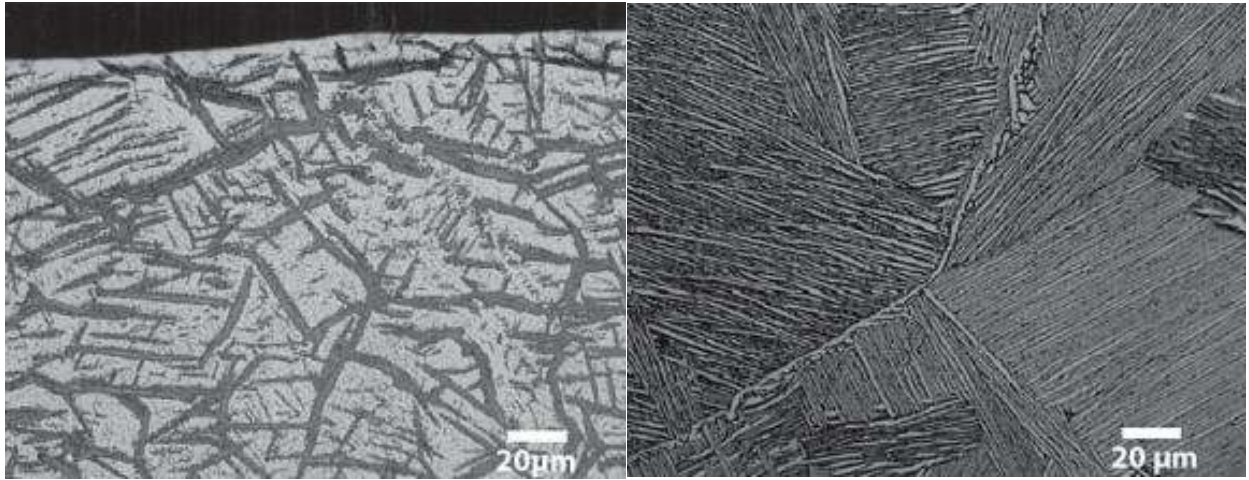


Figure 12 - Titanium Alloy Development [13].

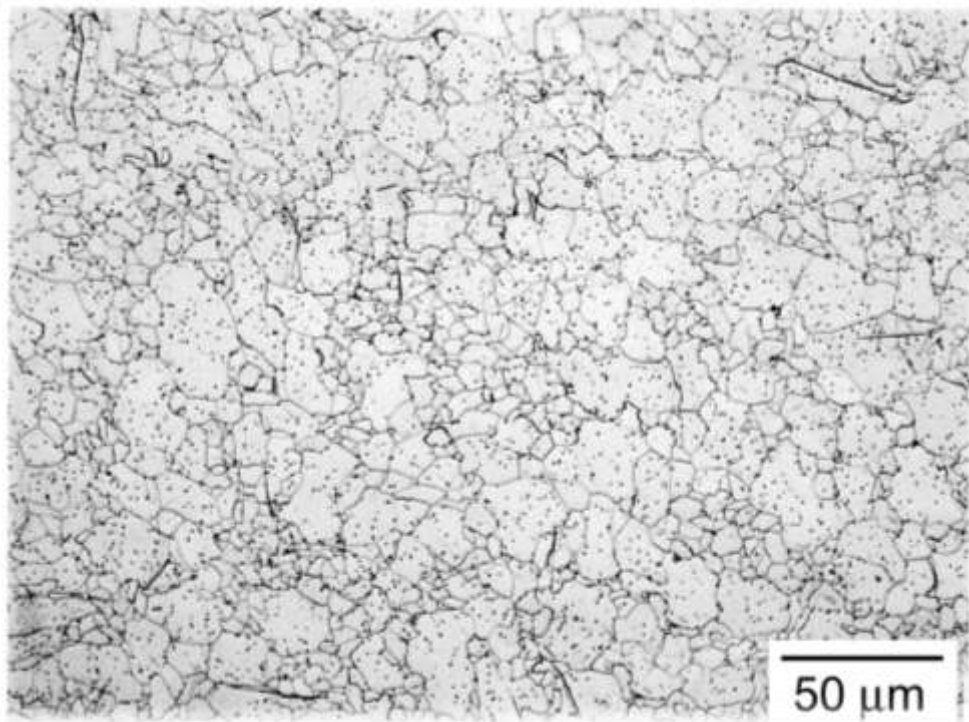


Figure 13 - Titanium Alloy Development [13].

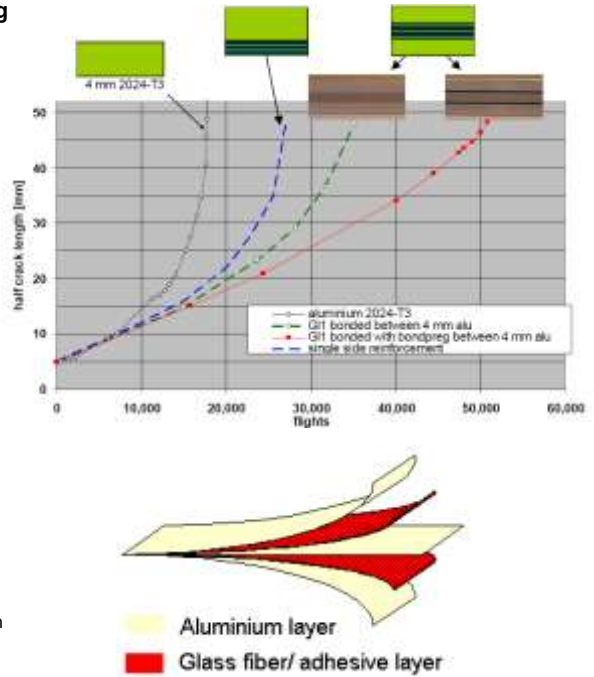
ber Metal Laminates (FML)

Glare is a sandwich material constructed from alternating layers of aluminum and glass fiber with bondfilm

- Weight reduction: 15 to 30%,
- Excellent Fatigue Resistance,
- Improved Impact Resistance,
- Excellent Fire Resistance Behavior,
- Lightning Strike Capability.



GLARE® shell with bonded stringers and doublers; A380 section 18, main deck panel



EADS Deutschland GmbH, corporate research center

Figure 14 - Glare Fuselage Panel Usage in A380 Aircraft [16], [17].

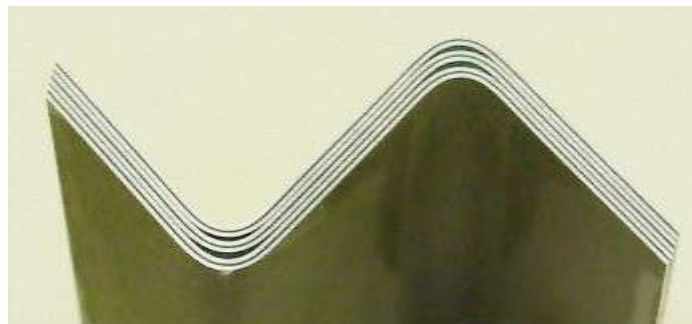
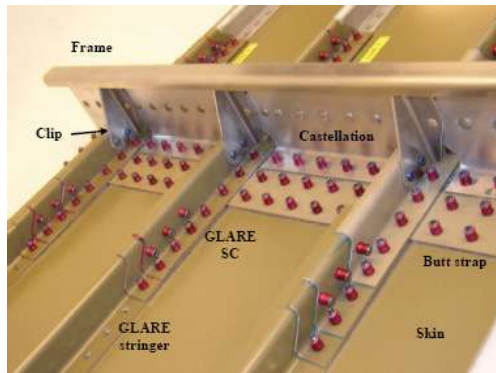


Figure 15 - Laminated Al components [18].

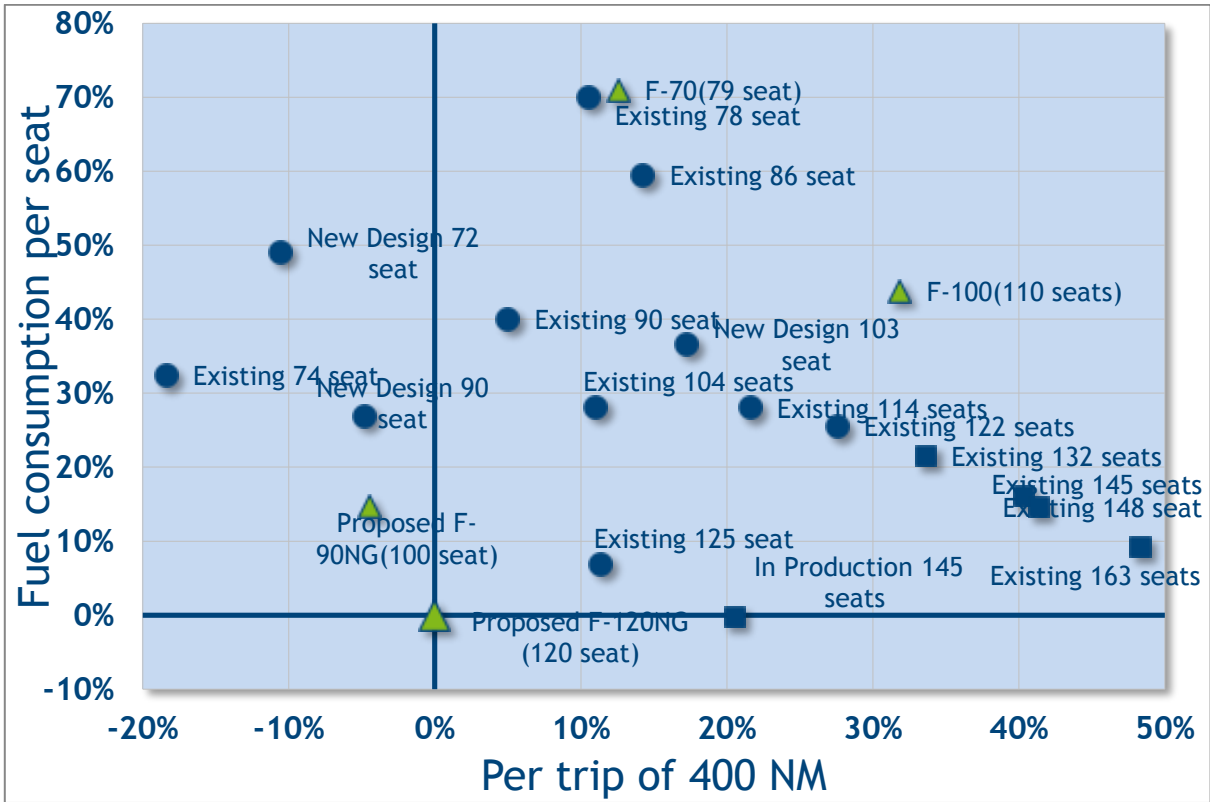


Figure 16 - Proposed F120 Aircraft to be developed as based on F100 (Authors are thankful to Mr. Rudi den Hertog and Mr. Maarten van Eeghen, NG Aircraft Company, Netherland for the information and the figure provided).



Figure 17 - 3-D Woven and Pi-Preform Joints and Large Integrated Composite Structure [5].

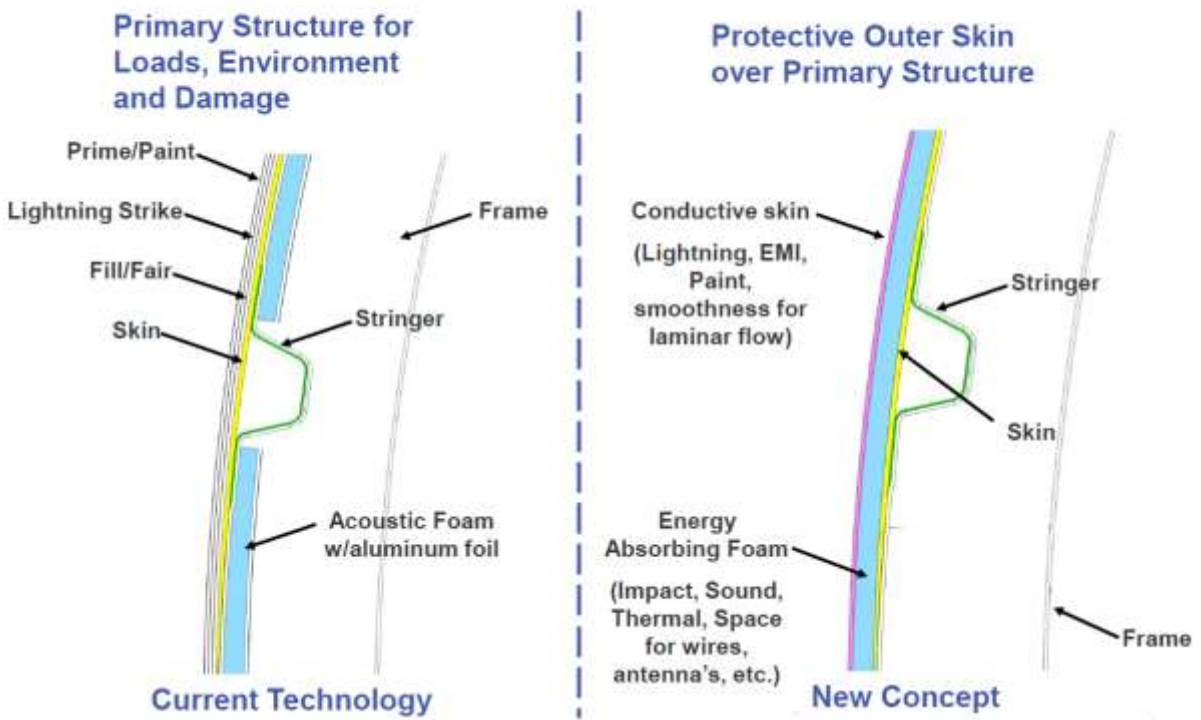


Figure 22 - New Composite Conductive Skin-Stringer Concepts with Energy Absorbing Foam [19].

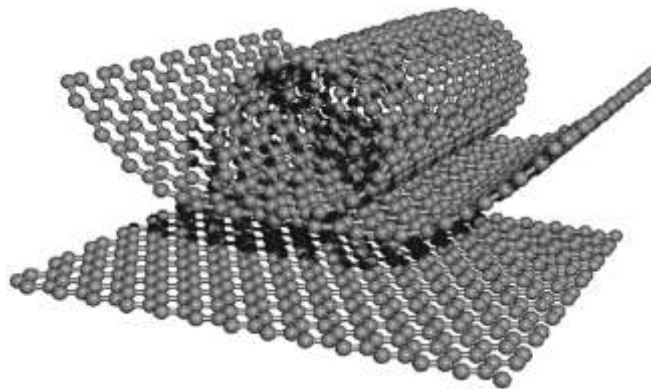


Figure 19 - Carbon Nano-tubes hexagonally shaped arrangements of carbon atoms bonded into a tube shape [20].

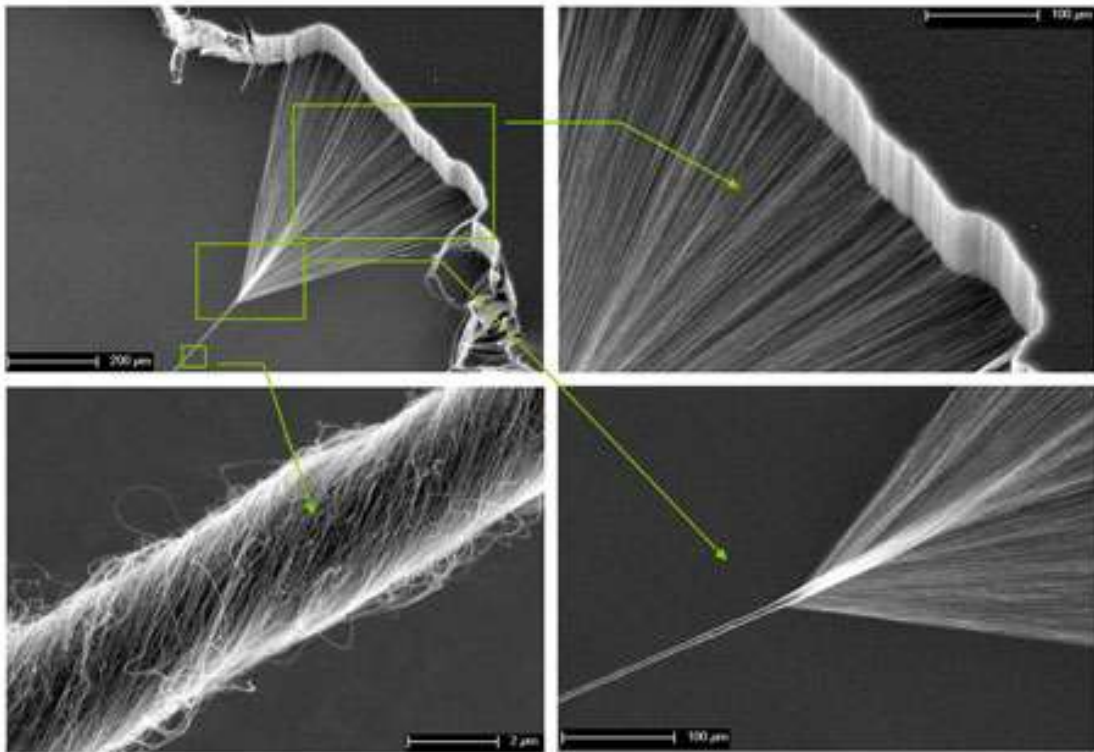


Figure 20 - From Nano-tubes to nano-fibers (Nanocomp Technologies Inc., Reference [20]).

Revolutionary High-Temperature Composite Materials

Revolutionary High-Temperature Composite

➤ to **425°C** for polymer-matrix composites (PMCs);

➤ to **1250°C** for metal-matrix / intermetallic-matrix composites (MMCs / IMCs); and

➤ to as high as **1650°C** for ceramic-matrix composites (CMCs).

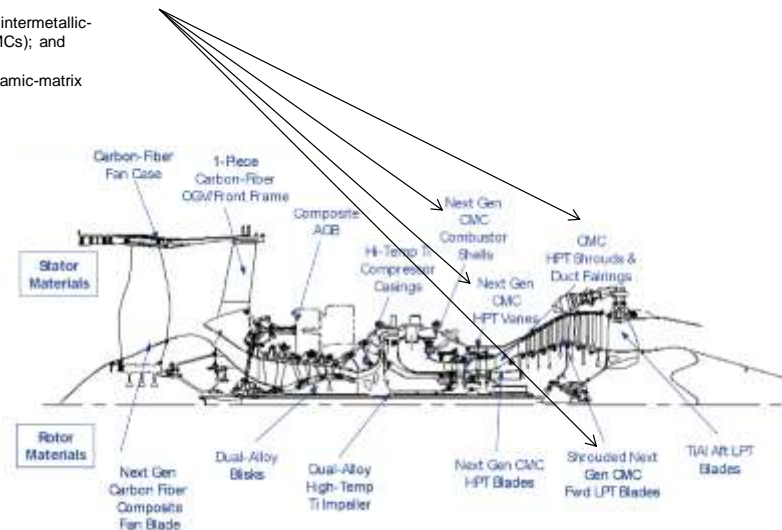


Figure 21 - Advanced High Temperature Engine Technology Development [23].

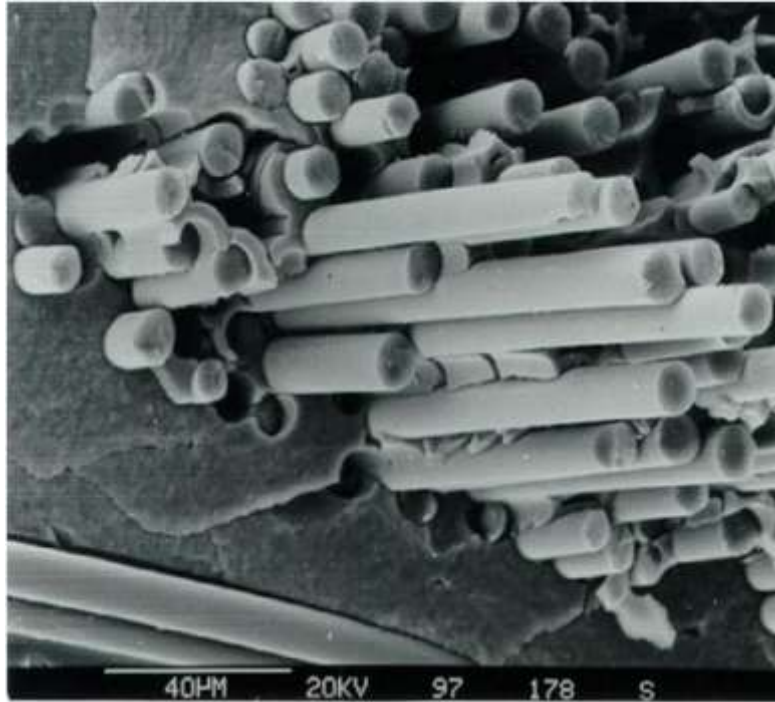


Figure 22 - Ceramic Matrix composites, ceramic composites reinforced with long or continuous ceramic fibers [21].

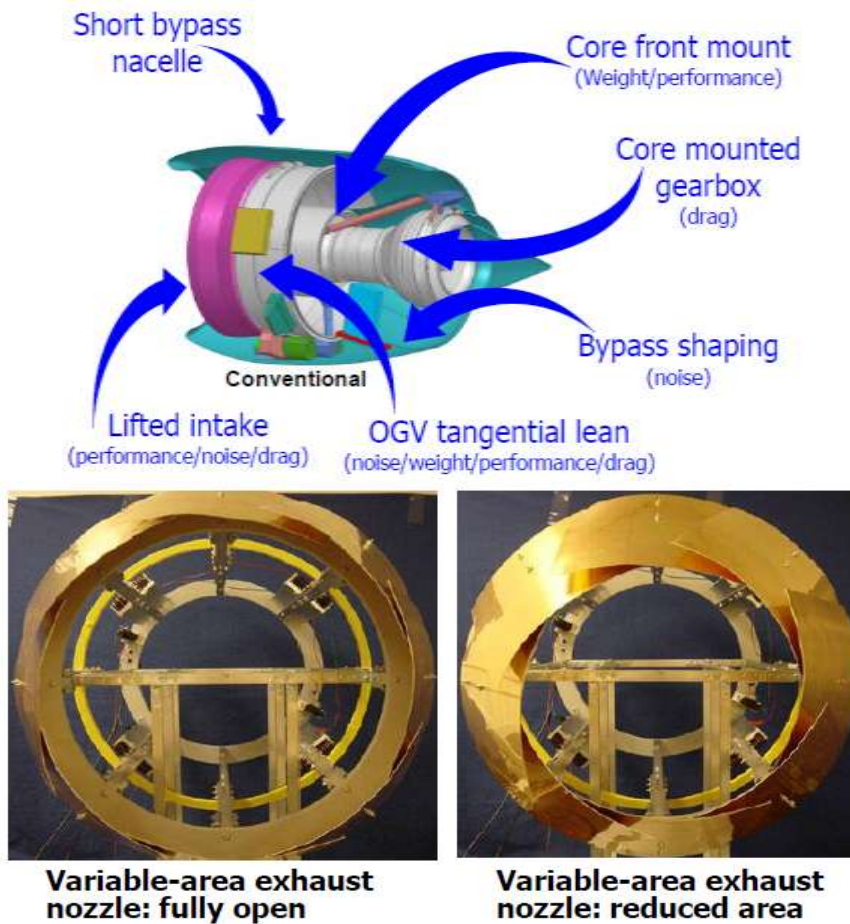


Figure 23 - Shape Memory Alloys (variable geometry nozzles for Light Weight Fan / Fan Cowl [5]).

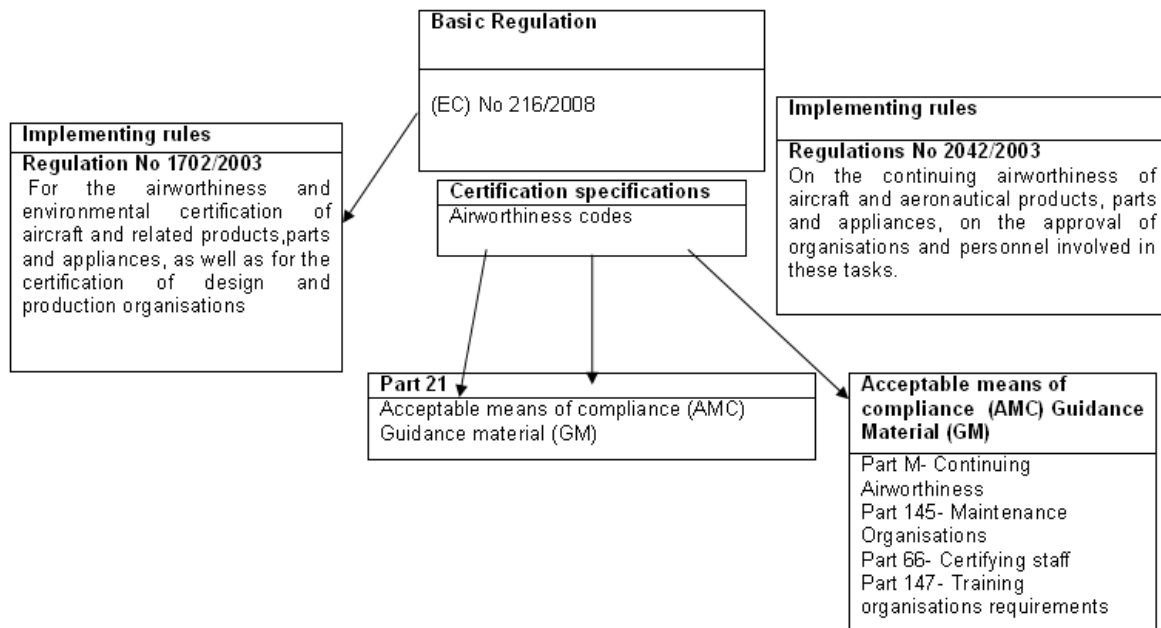


Figure 24 - Top Down Hierarchy of EASA Civil Aviation Regulations Covering the Complete Product Life Cycle of an Aircraft.

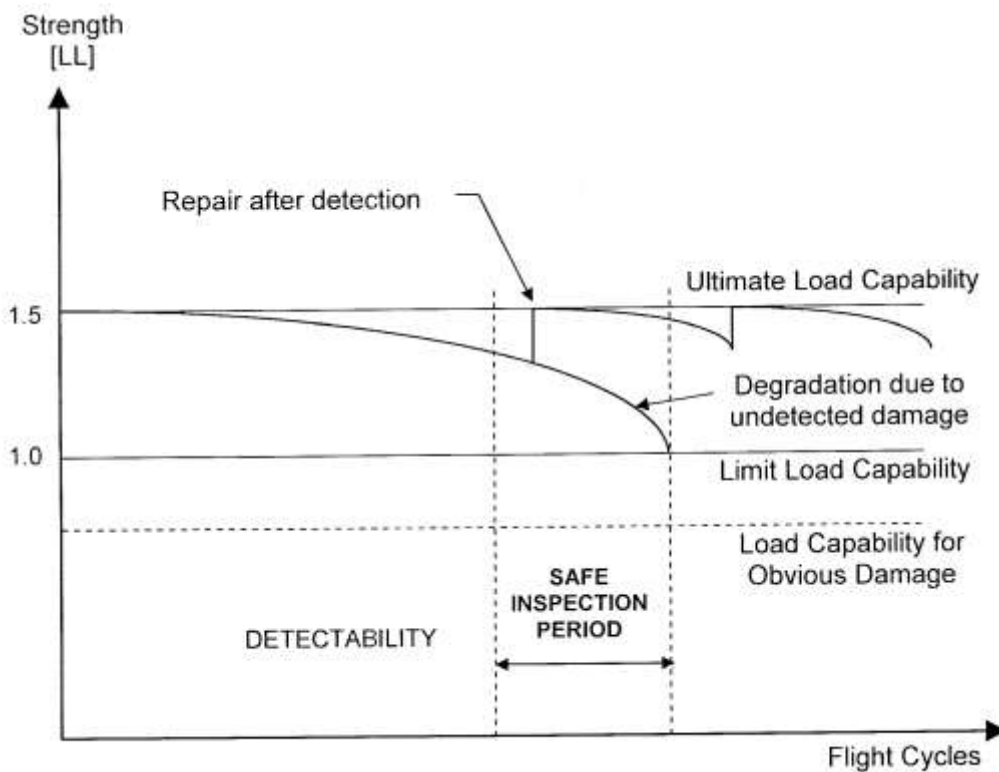


Figure 25 - Aircraft Structures Loading, Damage Detection, Inspection; Repair-ability Illustration.

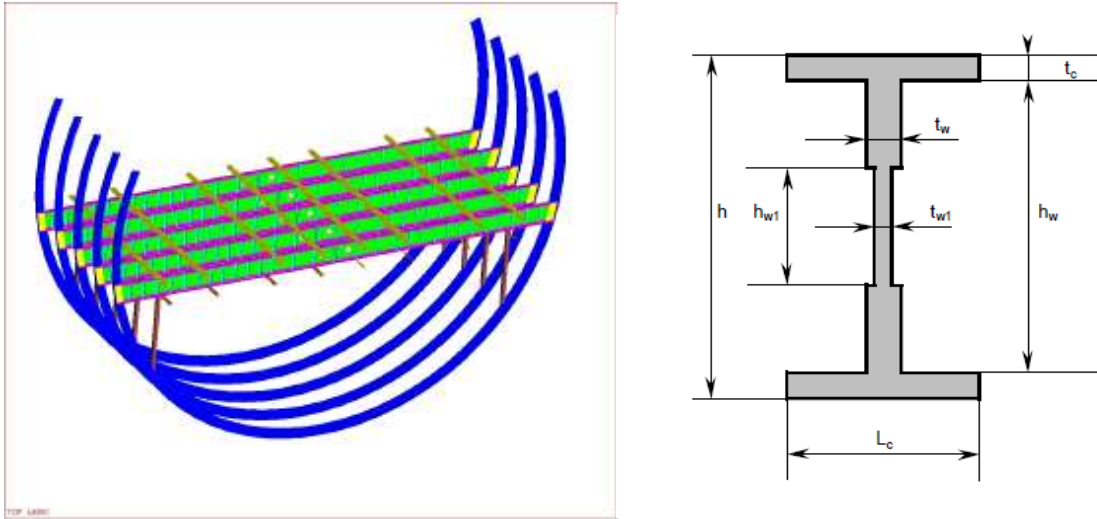


Figure 26 - 3-D view of The Floor Beam Structures and Cross Sectional Dimensions for Design Iterations [29].

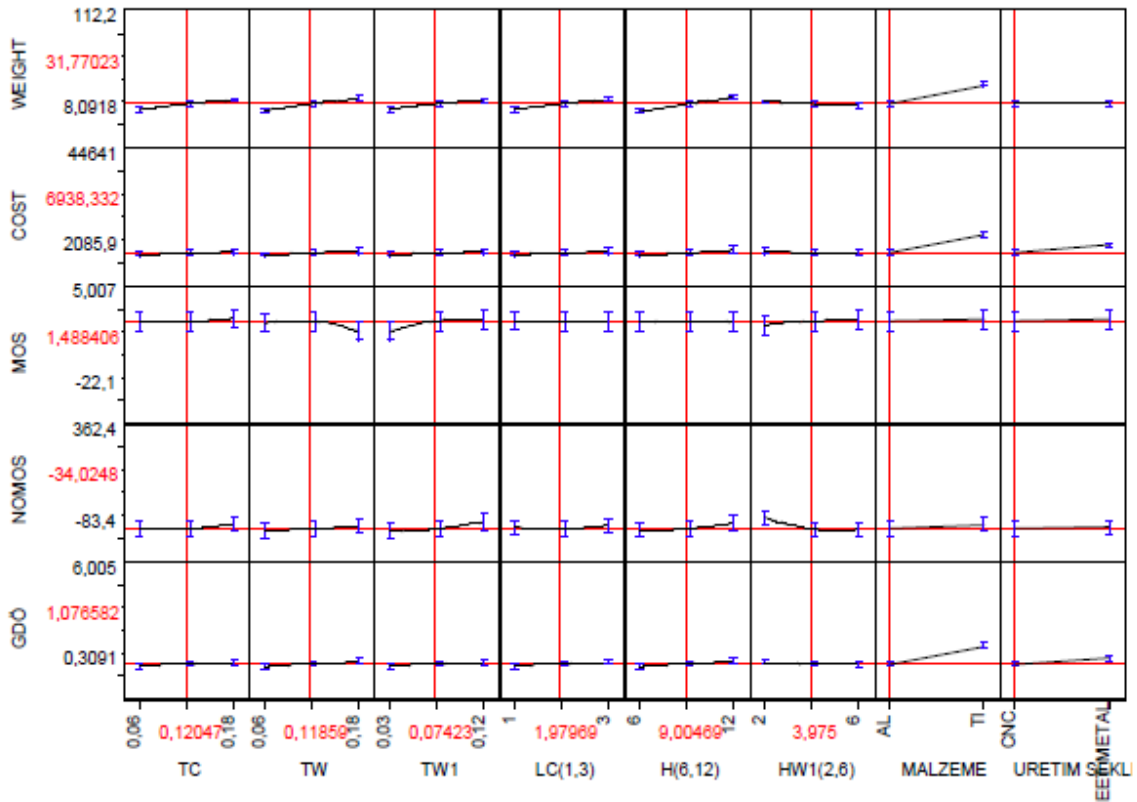


Figure 27 - Sensitivities and responses to the design variables for the floor beam design [29].

Acknowledgements

Significant part of this work had been accomplished while the first author was working as Manager of Design, Development and Projects and Head of Design Organization of EASA21J.418 at Turkish Airlines Technic. He is grateful to; Prof. Dr. Temel Kotil, CEO Turkish Airlines, and Dr. İsmail Demir, CEO Turkish Airlines Technic, Turkey for their encouragement for preparing this manuscript and sharing their very valuable comments throughout the work. Finally it should be acknowledged that this study would not have been possible without extensive supports of; Mr. Zeki Keskin, Structural Compliance Verification and Chief Engineer, Mr. Tarkan Sancaktar, Mr. Uysal Karlidag and Mr. Bilal Demir, Design Engineers at Turkish Technic Design Organization, Structural Engineering.

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Some Properties of Cu-B₄C Composites Manufactured by Powder Metallurgy

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Abstract

In this study, some properties of cold pressed Cu-B₄C composites were investigated. Commercial copper powders with 40 µm particle size were reinforced with B₄C in a 40µm particle size at ratios of 0, 1, 2, 3 wt.% for improving mechanical properties of copper used for electrical conductivity. Cu-B₄C composites have been fabricated by powder metallurgy method and sintered at 700°C for 2h in open atmosphere and then subjected to cold pressing following sintering process. The presence of Cu and B₄C which are dominant components in the sintered composites were confirmed by X-ray diffraction analyses technique and SEM-EDS. Scanning electron microscope (SEM-EDS) was showed that B₄C particles are distributed homogenously in the copper matrix. The relative densities of Cu and Cu-B₄C composites sintered at 700°C are ranged from 97.5% to 90.19%. Microhardness of composites ranged from 80.65 to 87.5 HB and the electrical conductivity of composites changed between 90.04 %IACS and 68.87 %IACS. It was observed that cold pressed Cu - 1 wt.% B₄C composites revealed promising physical properties.

Keywords: Cu-B₄C composites, cold pressing, electrical conductivity, powder metallurgy.

1. INTRODUCTION

In recent years, copper is widely used in industrial applications due to its high electrical and thermal conductivities, low cost (comparing with Ag and Au) and ease of fabrication. However, the relatively low hardness, low strength and poor wear resistance limit its extensive applications. These shortcomings could be avoided by the incorporation of ceramics into the copper matrix, such as oxides, carbides and borides [1, 2]. The incorporation of ceramic particulate reinforcement can improve the high temperature mechanical property and wear resistance significantly, without severe deterioration of thermal and electrical conductivity of the matrix [3]. Among the various ceramic particles, Al₂O₃, SiC and TiB₂ particles are commonly used. The particle reinforced metal matrix composites can be synthesized by such methods as standard ingot metallurgy (IM), powder metallurgy (PM), disintegrated melt deposition (DMD) technique, spray atomization and co-deposition approach. Different method results in different properties. The PM processing route is generally preferred since it shows a number of product advantages. Powder metallurgy process (PM) lends itself well for economical mass production components. Different metal matrix composites are manufactured by this PM route. The uniform distribution of ceramic particle reinforcements is readily realized. On the other hand, the solid-state process minimizes the reactions between the metal matrix and the ceramic

reinforcement, and thus enhances the bonding between the reinforcement and the matrix. However, the coefficient of thermal expansion (CTE) mismatch between the reinforcement and the matrix will give rise to high residual stress, which leads to the low tensile ductility of the composite [4]. Boron carbide (B₄C) cermets and boron carbide-based composites serve as promising materials for a variety of applications that require elevated mechanical properties, high neutron absorption cross section, high melting point, good wear and corrosion resistance [5, 6]. Boron carbide (B₄C) serves as a potential reinforcement for making composite material due to its high hardness (2900–3900kg/mm²), high neutron absorption cross-section and excellent thermo-electrical properties in addition to low density (2.52 g/cm³), high melting point (~2450 °C), high elastic modulus (448 GPa) and chemical inertness. B₄C high modulus ratio (1.8×10⁷ m) and preserved hardness even at temperatures above 1100 °C makes it as a strengthening medium in high temperature applications. These unique properties of B₄C at room and high temperature makes it a key material for various high technology applications, such as fast-breeders, neutron moderators in nuclear reaction, power generation in deep space flight applications, microelectronic, medicinal, light-duty bulletproof armors, blasting nozzles, abrasive water-jet cutting equipment, high temperature thermoelectric devices, high-temperature structural parts, cutting tools, rocket propellants, wear-proof parts and thermo-mechanical applications [7]. However, one drawback of

the boron carbide is lower thermal conductivity (TC). The research about improving the TC of the B₄C cermets and boron carbide-based composites is much less, two authors have previously fabricated B₄C/metal and B₄C/C composites to obtain high thermal conductivity materials, respectively [5].

In the present investigation, ceramic based B₄C is introduced to metallic copper to improve mechanical properties of copper.

2. EXPERIMENTAL DETAILS

Commercial copper with a particle size of 40 μm and SiC powders with a particle size of approximately 40 μm were used as starting materials. Commercial copper powders were reinforced with B₄C at ratios of 0, 1, 2 and 3 wt.%, respectively. These powder mixtures were compacted by uniaxial hydrolic press and then sintered at 700°C for 2h in open atmosphere for manufacturing composite samples. Then, sintered composites were exposed to cold pressing by uniaxial hydrolic press with a pressure of 180 bar. Following the manufacturing process, composites were analyzed by XRD technique using Cu Kα radiation with a wavelength of 1.5418 Å in order to determine the phases formed in the composites body. Microstructures of the samples were examined by Jeol LV6000 scanning electron microscope and Nikon Eclipse optical microscope. EDS analysis was conducted

to detect Cu, B₄C and possible copper oxides, copper-boron compounds within and at Cu-B₄C interfaces. The relative density of the composites was measured according to Archimed's principle, the microhardness and the electrical conductivity of both pure copper and composites were determined by Brinell Hardness with a load of 31.25 kg and GE model electrical resistivity measurement instrument. The results of electrical conductivity values were performed on the polished samples. The electrical conductivity of samples was determined by taking inverse of resistivity.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Optical micrographs of pure copper and Cu-B₄C composites sintered at 700°C for 2 hours were shown in Figure 1. As it can be seen in Fig., copper matrix is seen as light colored areas and black-cornered shapes denote B₄C particles. Reinforcement particles, It was observed that B₄C were homogenously distributed in the copper matrix. Copper grain boundaries are distinguishable from the microstructures after the etching of samples with nitride acid of 10% solution (Fig.1).

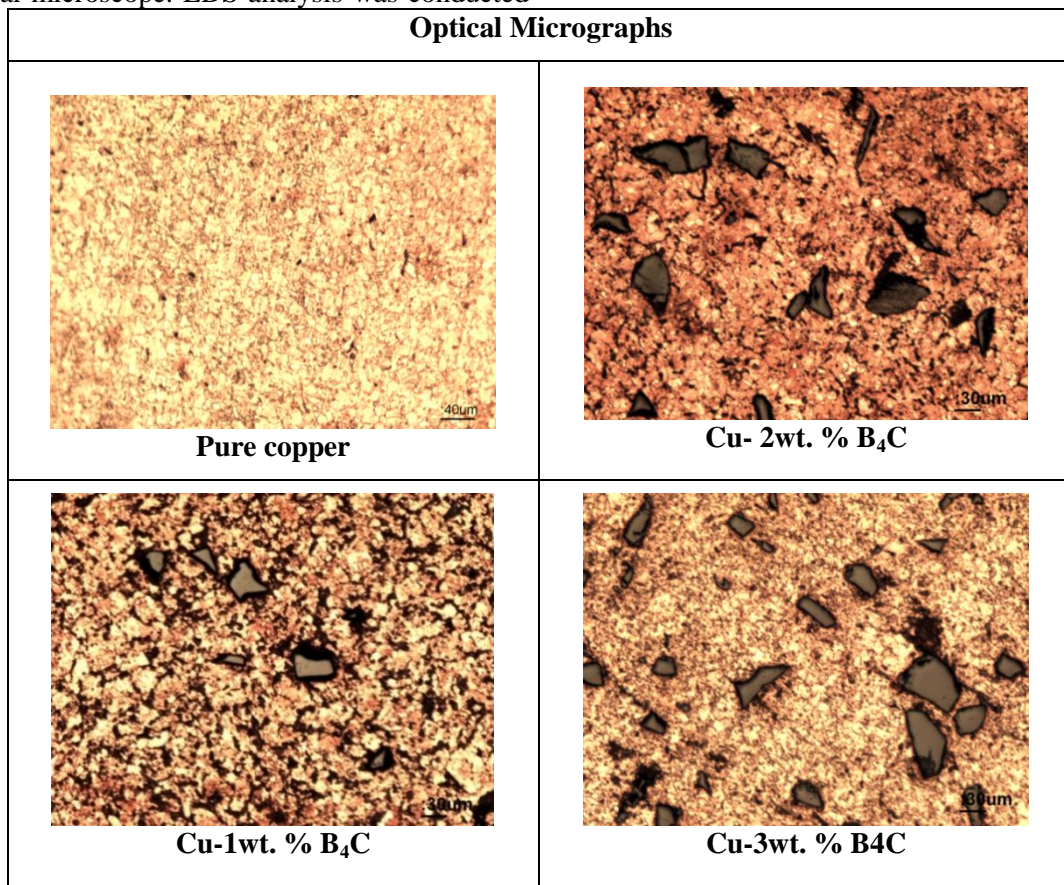
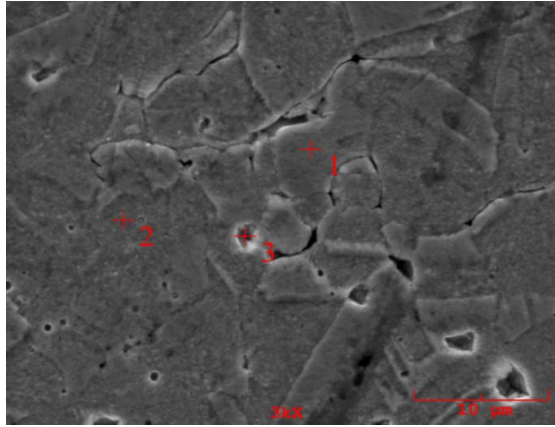


Figure 1 - Optical micrographs of pure copper and Cu-B₄C composites sintered at 700 °C for 2 hours.

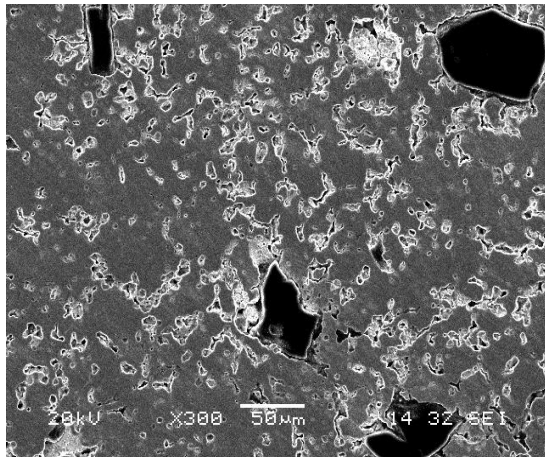
Figure 2a-c shows the SEM images with EDS analyses of the composite samples as well as pure copper. The dark, cornered shapes indicate B₄C and grey colored zones point out copper matrix as confirmed by EDS analysis (Fig. 2a-c). In the SEM images, white areas probably indicate alumina resulted from polishing and does not characterize any phase (Fig. 2b). Result of general EDS

analysis, there were small amounts of oxygen element in copper and composites samples (Fig. 2a-c). This was probably resulted from the oxidation of the matrix during sintering, however Al evidence as well as oxygen in the EDS analysis also indicated alumina remained from polishing process.



| wt. % | Marks | | |
|-------|--------|--------|--------|
| | 1 | 2 | 3 |
| O | 0.997 | 0.980 | 0.498 |
| Al | 0.309 | 0.118 | 0.435 |
| Cu | 98.694 | 98.902 | 99.067 |

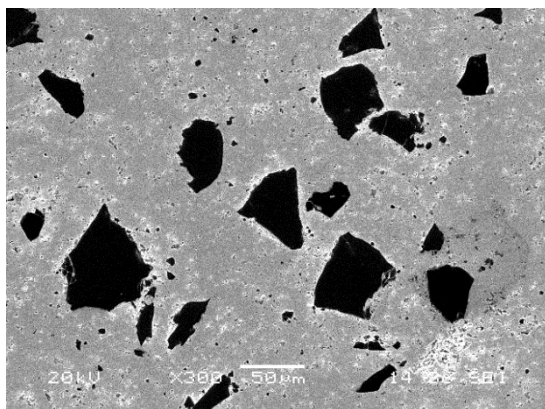
(a)



| Sample | wt. % | | |
|----------------------------|-------|-------|--------|
| | O | Al | Cu |
| Cu-1wt. % B ₄ C | 7.248 | 4.818 | 87.934 |

(b)

Figure 2 - SEM images with EDS analyses of samples sintered at 700 °C for 2 hours, (a) pure copper, (b) Cu-1wt. % B₄C, (c) Cu-3wt. % B₄C



| Sample | wt. % | | |
|----------------------------|-------|-------|--------|
| | O | Al | Cu |
| Cu-3wt. % B ₄ C | 2.570 | 1.790 | 95.640 |

(c)

Figure 2(continued) - SEM images with EDS analyses of sintered samples sintered at 700 °C, (a) pure copper, (b) Cu-1wt. % B₄C, (c) Cu-3 wt. % B₄C.

3.2. XRD Analysis

XRD analysis showed that no copper-oxide and B_4C phase, due to amount of B_4C in the samples possibly remained under the detection limits of XRD instrument, were detected and the dominant phase consisted of copper

(Fig. 3-4), SEM-EDS analyses revealed small amount of oxygen and evidence of B_4C phase were existed in the samples.

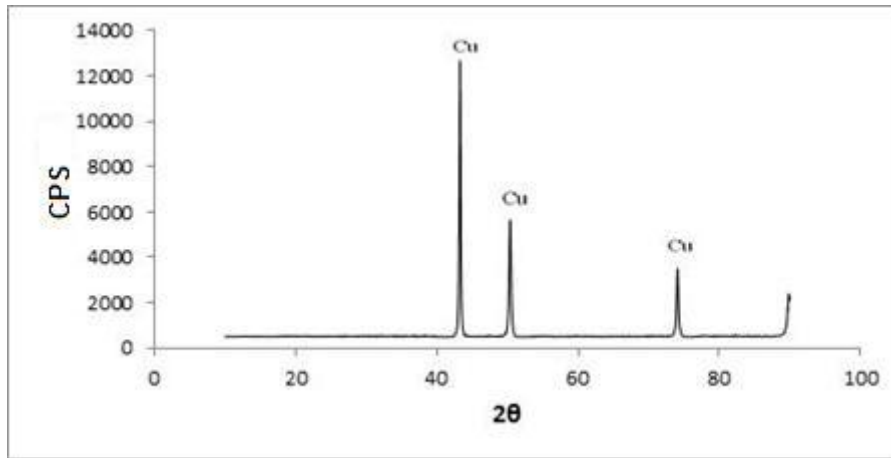


Figure 3 - XRD analysis of pure copper.

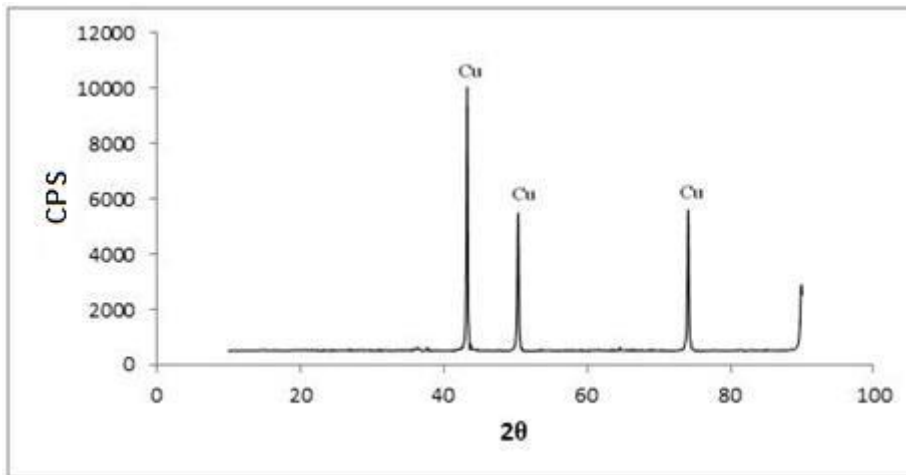


Figure 4 - XRD analysis of Cu – 3 wt. % B_4C .

3.3. Relative Density-Hardness-Electrical Conductivity

Relative density, hardness and electrical conductivity values of sintered and cold pressed commercial pure copper and $Cu-B_4C$ composites were given in Table 1. From the Table 1, the relative density and the electrical conductivities of the $Cu-B_4C$ composites decreased, while hardness values of them increased with the increment in the amounts of B_4C . Copper has high thermal expansion, while B_4C has low, by the result of this, significant amount of dislocation occurred because of great thermal expansion mismatch during sintering. Thus, by increasing amount of B_4C in composites, the hardness of the composites increased. Nevertheless, electrical conductivity of the samples decreased by decreasing the relative density because of lower density means higher porosity which acts insulation barrier for electron pass through between Cu grains [8]. It can be claimed that cold pressed $Cu - 1$ wt.% B_4C composites revealed promising

physical properties according to obtained results given in Table 1.

Table 1 - Relative densities, hardnesses and electrical conductivity values of cold pressed copper and $Cu-B_4C$ composites.

| Properties | wt.% B_4C | | | |
|--------------------------------|-------------|-------|-------|-------|
| | 0 | 1 | 2 | 3 |
| Relative density, % | 97,5 | 92,15 | 91,15 | 90,19 |
| Hardness, HB | 80,65 | 81,7 | 82,8 | 87,5 |
| Electrical conductivity, %IACS | 90,04 | 80,91 | 69,1 | 68,87 |

Acknowledgements

Authors thank to technician Ersan Demir of Sakarya University for his contributions during this study.

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Continuous Quality Improvement in Textile Processing by Statistical Process Control Tools: A Case Study of Medium-Sized Company

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Abstract

Maturity of companies, organizational culture and costs are some of the major limiting factors for implementation of cost-effective six sigma methodology in Bosnian companies. Purpose of this paper is to analyze possibilities of applying six sigma concept in a medium-sized company with 200 employees. Considering all characteristics of the company, in this case, implementation model was proposed and tested. The project included process monitoring using statistical process control charts for two different periods, before and after improvement. Dominant defect and its causes were identified and it was found out that the process was out of control. Implementation of improvement measures, dominant defect was eliminated but the process has remained out of control. As a conclusion, the test indicated that the model is effective but it takes more iteration to achieve the desired state.

Key words: Six Sigma, process improvement, defect, control chart.

1. INTRODUCTION

Six Sigma as more advanced quality system has recently become attractive to many companies especially for those that aspire to business excellence. After many successful stories of implementing Six Sigma in large global companies such as General Electric, Caterpillar and others, today Six Sigma is more likely becoming a successful story in some smaller companies over the globe [1].

Also, there is no unique definition of Six Sigma, but it is possible to outline some positive aspects. Six Sigma utilizes hardware and software of information technology for problem solving [2]. It is a platform for self-assessment and improvement, on a project-by-project basis using statistical tools for real-world applications by specially trained personnel [3]. Six Sigma is a more advanced level of quality, which will certainly implement those organizations that tend to business excellence after QMS certification per ISO9000 series. There are four elements of the Six Sigma definition: parallel-meso structure, improvement specialists, structured method, and performance metrics [4]. Six Sigma is not a certification scheme for quality like ISO9000 or ISO14000. There is no organization that claim to be 'certified to Six Sigma', and there is no internationally recognized body to certify or register companies that are 'Six Sigma compliant' costs [5]. Six Sigma is an opportunity for organizations to increase the

product/process quality and profits, and to reduce costs [6].

In statistical terms, sigma (σ) is a measure of scattering processes. It is defined as having less than 3.4 defects per million opportunities or success rate of 99.9997%, which is the ultimate goal to improve and reduce the costs of poor quality at $1 \div 2$ % [7]. Six Sigma is defined as a set of techniques based on Statistical Process Control (SPC), which can help companies to achieve significant improvement in quality and therefore increase competitiveness [8].

Six Sigma provides a comprehensive plan, which helps companies to integrate appropriate statistical tools and other techniques in a "comprehensive" tool, for process improvement. These tools can be applied in individual phases of Define, Measure, Analyze, Improve and Control (DMAIC) methodology in order to establish an effective processes quality improvement system [8] [9]. This structured method is based on Plan, Do, Check, Act (PDCA) cycle, but Six Sigma specifies the tools and techniques to use within each step, which is unique to Six Sigma [10]. The six sigma achievement also depends on the production age [11]. Therefore, following six sigma methodology this research will not attempt to hit the six sigma target. Firstly, the aim of the research is developing and testing a model for defect monitoring in a textile cutting process based on six sigma methodology. Secondly, testing the model for the

six sigma methodology implementation to optimize a textile cutting production process in a mid-sized company. With the aim of showing state of the process, the data collection was carried out during two periods, i.e. before and after correction measure implementation. The length of the pre-test period was about one month and 168 batches have been checked. The length of the test period after implementation of the correction measures was approximately 30 days and also 154 batches (or 642,636 pieces) have been checked. In the discussion below, results of the analysis for considered periods are presented for three shifts [12].

Implementing Six Sigma in small and medium-sized enterprises is not sufficiently investigated and there is not enough data about successful implementation. The traditional approach to the implementation specific for large companies is expensive and requires a lot of resources. Therefore, implementation of Six Sigma in small and medium-sized enterprises represents an additional barrier to the success of such projects [13].

Six sigma concept discussed in this paper is developed for an export-oriented medium-sized company that employs about 200 workers, which took in account all specifics of business processes and problem faced. The approach in the implementation typical for large companies was modified in a manner that will enable the implementation of projects using fewer resources, which makes it applicable to medium-sized companies [14], [15]. In order to maintain its competitive advantage, the company is dedicated to the programs of implementing advanced quality systems such as six sigma or integrated lean six sigma, immediately after implementation of ISO standards [16].

The purpose of this work is to develop and test model for textile cutting process optimization suitable for medium-sized company. The model can be considered as framework for the widespread use of six sigma in Bosnian companies, particularly in SMEs. The primary goal of the test is to reduce the number of defects that occur in the process. Due to defects in the production process, outages had been observed that lead to delay delivery in some cases. Therefore, additional time, material and transportation are necessary in order to deliver parts to the customer site per Just in Time principle. In the worst case, this leads to reducing regular production time, working under stress, making extra costs, new defect accumulations, which reflects to the regular production process schedule.

2. RESEARCH METHODOLOGY

As a first step in developing model proposal was the approach to the production process mapping and categorizing on the major and side processes (subordinate). Also in this step was developed continuous improvement algorithm proposal and process

of nomination and selection of new projects. The second step was the identification of processes that have a determinate process of work, which is one of the main requirements for DMAIC methodology application.

The third step was the analysis of collected data from the process collected over past year and dominant cause identification using statistical tool. Also, in this step were tested process capability indices (C_p and C_{pk}) as indicators of process capability to produce output within specified limits. To get an impression of how well the process fits within the given specification range is represented by process capability index C_p .

$$C_p = \frac{USL - LSL}{6\sigma} \quad (1)$$

USL – Upper Specification Limit

LSL – Lower Specification Limit

σ – standard deviation

C_{pk} index shows how well the process is centered within the specified limits.

$$C_{pk} = \min \left[\frac{USL - \mu}{3\sigma} \quad \text{or} \quad \frac{\mu - LSL}{3\sigma} \right] \quad (2)$$

μ – process mean

Calculation and interpretation of these indices included the following assumptions:

- data distribution can be approximated by a normal distribution;
- the process is stable and causes no significant variation (under control);
- process tracking using appropriate control charts (single-sided specification).

Some companies require that the minimum value of C_p index is 1.33. Some other companies raise this requirement up to 1.67 or $C_p \geq 2$, what corresponds to the Six Sigma level. If $C_p < 1$, it means that process mean μ is outside of specified range and produces high percentage of defects but if the process is centered in that case $C_{pk} = C_p$, and if $C_p > 1$ then process meets specifications. Also, if the process is "under control" computing capability index does not make sense [15].

The fourth step was the improvement measures and result verification. As Six Sigma analysis tools fishbone diagram for brainstorming sessions, p-control chart, Pareto analysis and correlation are used in this step [14]. For the result verification software is used for readings of attribute control charts and capability indices calculation and Pareto.

2.1. Structured Method

Once the improvement project is implemented, the performance of the process is necessary to improve continuously per DMAIC methodology with the aim of business excellence aspiration. This approach allows the

effective use of data in order to eliminate the causes of defective products. In accordance with this requirement, the DMAIC methodology application flowchart is designed for the manufacturing process *Figure 1*.

Process monitoring control charts are used to collect and analyze data, and based on that, it is possible to act proactively in problem solving. Using p -control chart it is possible to track "good/bad" part characteristics. Using $\bar{X}R$ -control charts it is possible to track measured values of the part features. Fishbone diagrams are used for brainstorming sessions that take into account the

main affecting elements of the process quality (man, machine, material, method, environment, and measurement). If any of these elements have significantly changed, the system of the processes will be changed as well, and there will be corresponding changes in product quality.

Software is used to analyze collected data that automatically show the state of process in the control charts and calculating process capability indices C_p and C_{pk} .

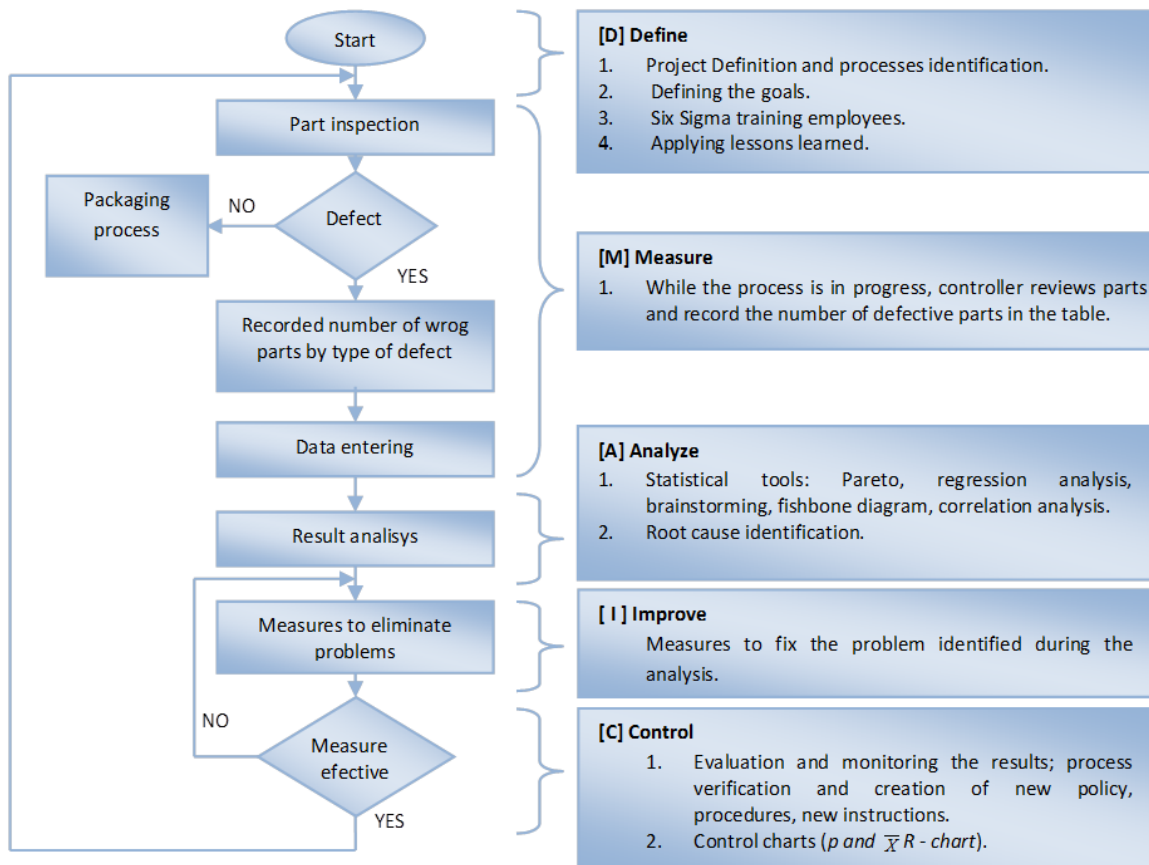


Figure 1. Continuous improvement framework flowchart

2.2. Strategic Project Nomination and Selection

Project approach guarantees a well-founded basic cause analysis and thus optimization sustainability. It is disciplined process that will help managers to focus on developing and delivering improved products and services. The main idea is to build a system for defect monitoring and figure out how to eliminate them trough implemented projects [17].

The process of nomination and selection of projects is carrying out per the following algorithm (*Figure 2*). Everyone in the company is eligible for submitting project ideas and according to customer needs project of interest will be selected. Project selection process includes idea recognition, evaluation and finally selection. After this, according to the project complexity team can be assembled.

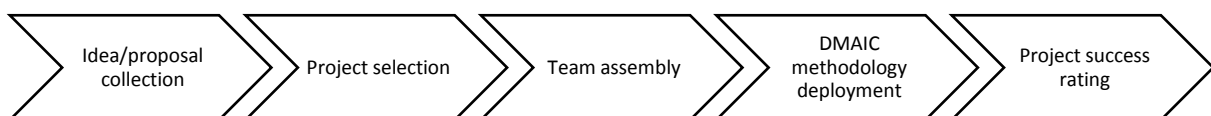


Figure 2. Strategic project selection and implementation

2.3. Improvement Specialists and Metrics

Due to the size of the company, it is anticipated that the project teams work "part time" on the project, and to be supervised by a Black Belt improvement specialist, who will manage projects and train team members [18].

Process performance will tend to improve with the use of the Six Sigma structure to reduce variation in organization processes by using Black Belt improvement specialists, a structured improvement procedure, and Six Sigma performance metrics with the aim of achieving strategic goals [19] [20]. Impact of various Six Sigma elements on project success within different project contexts such as parallel structure, improvement specialists, structured method, and clarity of performance metrics can together determine the success of process-improvement projects. Leadership engagement, strategic project selection, and psychological safety can positively affect these elements, on different ways in different project environments. Also, small and mid-sized companies have a low level of Six Sigma maturity, which could be successful in executing their project [21] [22].

Using this model, the company has a framework for optimizing textile cutting process, where it is necessary to take corrective action first in order to stabilize the process and then undertake improvement steps.

3. RESULTS AND DISCUSSION

According to the algorithm *Figure 1*, in this section is applied DMAIC methodology step by step as described above.

2.4. Process Mapping

The main production process of textile cutting is carried out in four phases and consists of nine CNC machines. Features of this process are high flexibility, high production, a large number of defective units, and longer production time per unit. On that basis, the main process operation had been identified in the following stages (*Figure 3*).

A side process of the textile cutting is "cutting through" (press cutting) that takes place in four phases and consists of three presses. Features of the process are high speed per unit of the product, a low number of defective pieces, modular parts (inflexibility) and low productivity.

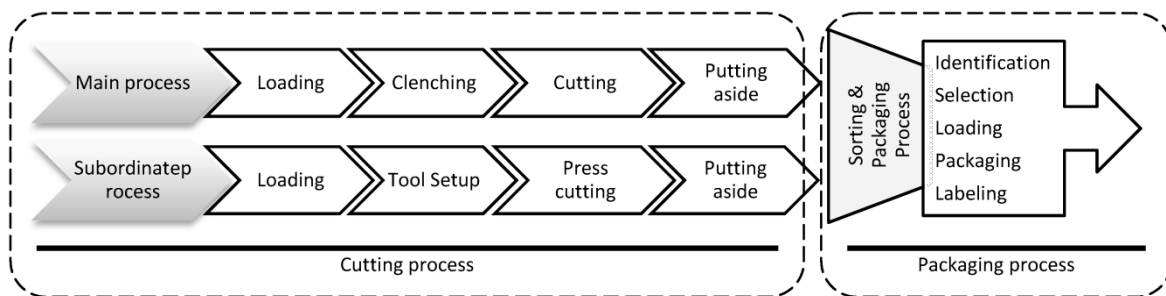


Figure 3. Textile cutting process

Another side process of sorting and part preparation for the next process of sewing had been identified as well. This process consists of selection and packaging of the parts per sewing plan. Features of this process are: small available space, slow identification and large variety of parts. On basis of sewing plans, workers pack required quantities of assemblies and sub-assemblies. Dominant defects from this process are wrong quantities and wrong parts, what is caused by workers. The previous three processes represent manufacturing process of textile cutting in which defects occur [12].

3.1. Project Selection

Statistically speaking, it was shown that the main process contributes most to the production of defective units and therefore according to the *Figure 2* this process is selected as an experimental project No. 1 for the optimization. The scope of the experimental project

included project definition and DMAIC methodology application, where was done the following:

1. Existing data analysis over the past 12 months;
2. Dominant defect identification;
3. Identification of main defect causes;
4. Correction measure proposals and implementation;
5. Measurements of process after improvement and its analysis.

Experimental testing of the model was carried out on the main textile cutting process for three shifts. Attribute characteristics of the process output were monitored using *p*-chart. For this purpose all parts were inspected, defects were recorded, and process indicators were calculated. The process consists of CNC machines and has the following stages: textile loading, vacuum clenching, textile cutting and putting aside (*Figure 4*).



Figure 4. Production process line

The material is loaded in multiple layers using a vacuum clenching to the bench. The knife cuts at the same time all loaded sheets of the material per CAD generated contour. In case something goes wrong during the cutting operation, the knife will not produce only one

defect but just as many as the sheets of the material is loaded.

3.2. Process Measure

While the process was running controller was monitoring the process and inspecting the parts using *p-chart* to track percentage of defective parts per batch. Figure 5 shows the state of the process for both periods.

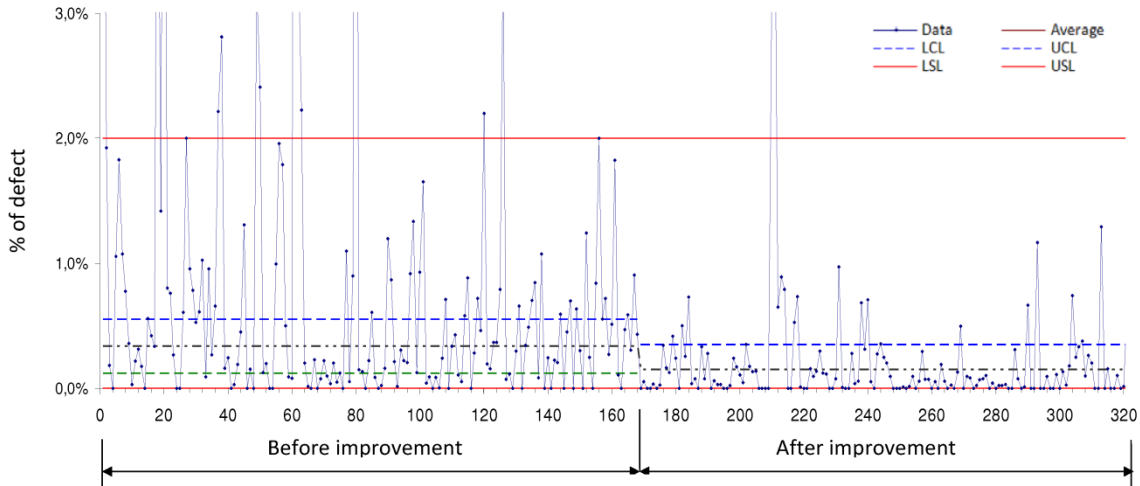


Figure 5. Process before and after changes

The process is not under statistical control because it does not operate within its natural process limits i.e. Upper Control Limit (UCL). Upper Specification Limit (USL) for the process is 2%, which in this case is not satisfied also. The process is very unstable and it is necessary to stabilize process first in order to obtain realistic values of process parameters.

histograms do not correspond to the normal distribution; it is more asymmetric distribution, i.e., the binomial distribution, which is characteristic for the attribute quality characteristics, which is the case here. In the period before improvement, histogram shows that only 64 batches (out of 168) have 0.2% of defects and 30 batches have 0.4% of defects. Also, the rest of the batches have some percentage of the defects.

3.3. Histogram Analysis

Figure 6 shows the defect histograms for both periods before and after improvement measures. These

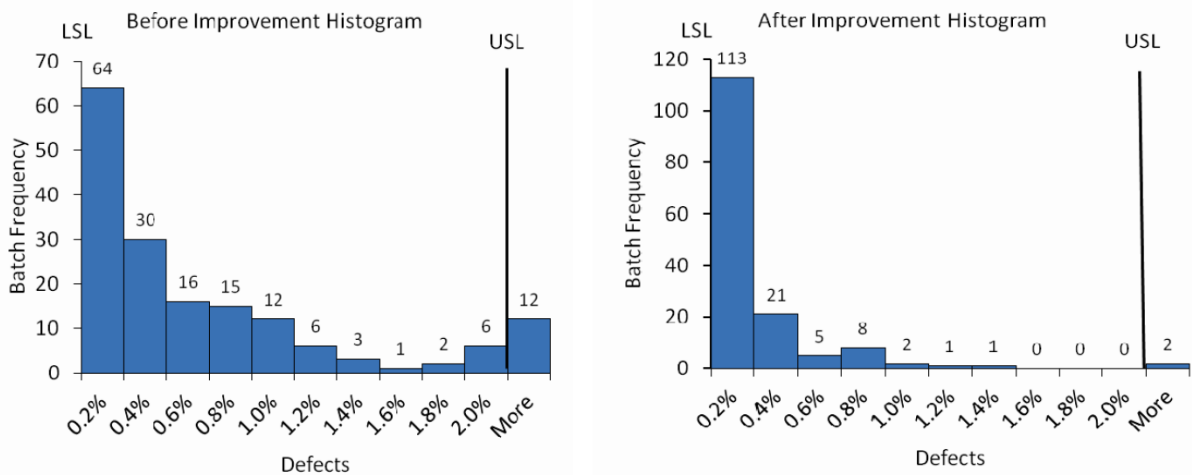


Figure 6. Histograms before and after improvement

Comparing with the period after improvement, histogram shows less spread of the defect through the batches. This improvement is significant because 113

batches (out of 154) had only 0.2% of defects, 21 batches had 0.4% of defects. Also, there are no batches with 1.6% to 2% of defects.

Outliers are evident in both cases. In the first period there were 12 batches outside USL which makes the process unstable. In the period after improvement outliers are still evident and there were two batches over 2%, which make the process unstable. So, after first iteration and dominant defect reduction process improvement is obvious but still unstable.

3.4. Discussion of Process Capability Indices

Based on calculated index value of $C_p = 4.64$, it is not possible to determine process sigma level. On the other hand, if PPM value is observed, which is 3352, that means that the process is about 4.2σ . This discrepancy between the PPM and C_p is explained by the fact that the process is not under control and that the values of process capability indices are meaningless. The value of standard deviation ($\sigma = 0.00072$), is much higher than in the other case for the period after improvement.

A similar comment applies to the period after improvement (*period II*), where $C_p = 5.58$, and value of PPM is 1491, which means that the process takes about 4.5σ level but still is not under statistical control. Thus, in both cases, none of the above process capability indices are unacceptable. This means that is necessary to stabilize the process first and then make other improvements. The standard deviation in this case is smaller than for the period before improvement (*period I*) ($\sigma = 0.000597$), which can be visually inferred from the control chart. Therefore, the process capability index values are unrealistic because the process is not under statistical control.

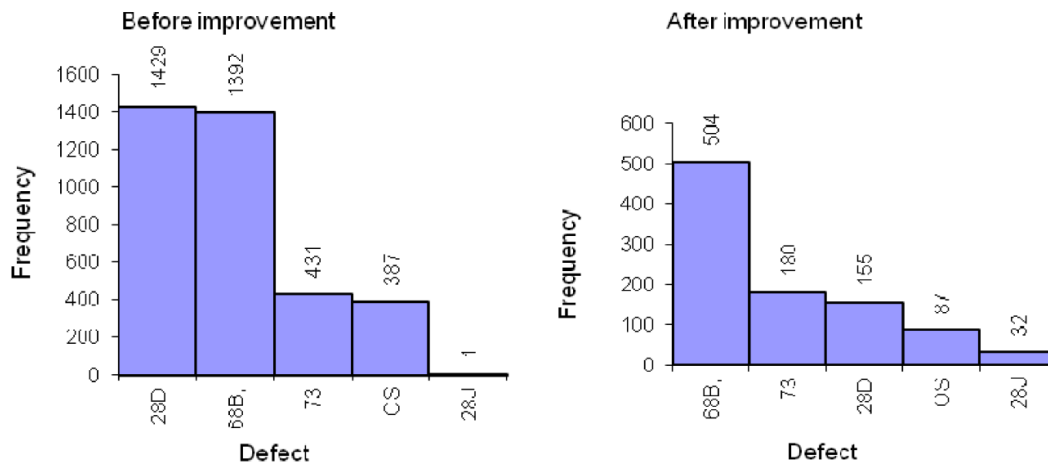


Figure 7. Two-period defect analyses

Another reason for the drawing thread is the blunt knife tip, where the first knife sting into the material and usually draws material thread. The cause of this bluntness is first sting knife tip hitting to knife guides or workbench in some cases.

3.6. Number of Defects and Batch Size Correlation

There was very small correlation between the batch size and number of defects (*Figure 8*), which means that little

In both considered periods there are outliers that appear to deviate markedly from the normal process flow. These extremes are usually caused by people not by machines. The actual value of the standard deviation will be smaller if outliers are neglected.

3.5. Pareto Analysis

Pareto diagram and regression are used to analyze collected data where interdependence between number of defects and batch size is explored. *Figure 8* shows defect frequencies from the process for both periods with the following meaning:

- Pulled fabric thread (defect code "28D")
- Marked defect on material (defect code "73"),
- Wrong cut - holes, „V-cut“, notch (defect code "68B")
- Dirty (defect code "28J"),
- Uncategorized defects (defect code "OS").

Pareto analysis showed that dominant defect for pretesting is pulled fabric thread "28D" (*Figure 8*). After brainstorming session and problem solving procedure, defect 28D is reduced. This is systemic defect caused by machine due to irregular sharpening of material cutting knife (*Figure 9*). Due to bumps on the knife cutting edge, formed by sharpening grinding wheels, the knife couldn't cut the last fabric thread on the cutting contour, causing drawn fabric thread in the next step "putting aside" (see *Figure 4*).

propagation of defects is evident from the increase in the number of material sheets of the batch. That means, if machine cuts 20 sheets at once, and if the operator mistakenly put an improper diameter drill, the drill will go through all 20 sheets and thus produce at least 20 defects. In that case, defect propagation is much higher than with a smaller number of sheets. Correlation was slightly higher for the period of "I" and was $r = 0.38$, while for the period of "II" correlation coefficient was

smaller $r = 0.21$, which means that none of the calculated values are statistically significant and therefore they are

not further discussed.

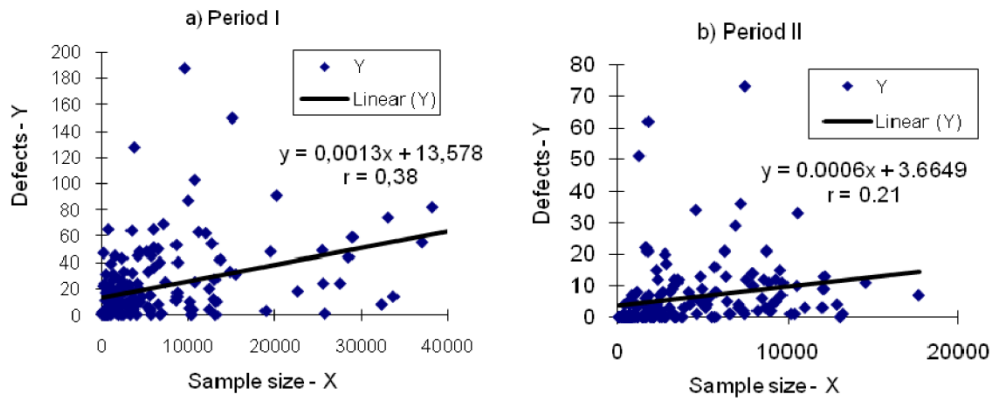


Figure 8. Defects depending on the batch size

As a next step it is necessary to maintain statistical process control per DMAIC methodology principle. Based on the previous iteration results it is necessary to take new actions in order to stabilize the process and then continue with the improvements that lead to six sigma quality level. This includes evaluation and monitoring of the each previous phase results, process verification and modification and creation of new policies, procedures, instructions to the employees. Software for defect monitoring in the production process

provides a clear summary report of all process parameters creating a good history record.

3.7. Improvement

Pulled fabric thread defect is solved by changing method of knife sharpening, as a result of brainstorming. The new sharpening method uses a fine sandpaper belt instead of grinding wheels, which is incomparably much more convenient for setting grinder and making knife cutting edge uniform.

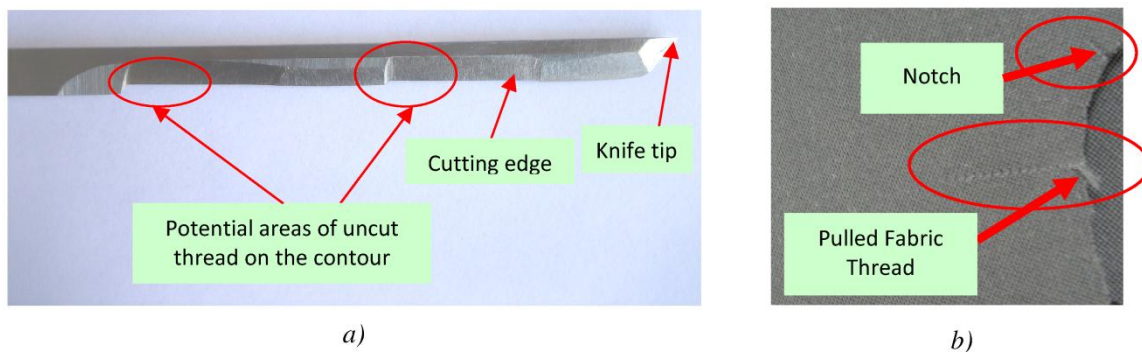


Figure 9. Samples: a) Irregular knife sharpening; b) Pulled fabric thread "28D" and notch "68B"

With these changes the dominant defect "28D" (pulled fabric thread) is significantly reduced. Defect "68B" (irregular cut) caused by human factor became a new dominant defect. The process could be under control if the defect "68B" is neglected. In that case process picture becomes quite different, and process capability index becomes acceptable.

of quality characteristics such as the depth of the notch, the position of the hole, "V cut" etc. Based on **Figure 1**, it is necessary to maintain statistical process control, and in accordance with the results of the process monitoring to undertake actions. Software for defect monitoring gives a concise and clear picture of all the process parameters.

3.8. Control

Process control includes the evaluation and monitoring of the previous stage results, verification of the process modifications and creation of new policies, procedures, and instructions to the employees. In this phase basic tools are control charts: p-chart for qualitative characteristics and \bar{X} R chart for quantitative monitoring basis in decision-making process to eliminate defects. Systemic and systematic approach can be applicable to

4. CONCLUSION

The main result of applying the described method can be suitable solutions applicable to small and medium-sized organizations and using project part time workers. Application of the algorithm promises success in improving the process because it provides a scientific

the entire production process, allows that in any moment the process parameters can be easily calculated (natural

process limits, standard deviation ...), and process capability indices C_p , C_{pk} . On that basis sigma level of the process can be determined. Software enables company managers to have in one place all information's about the process. Accordingly, it is possible to take appropriate corrective and preventive actions.

Proof of this is an iteration of the experimental testing of the concept in order to evaluate the model efficiency and influence of certain factors on the process stability. In

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Modulated (Spinodal) Alloys

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Abstract:

In this work, after defining spinodal reactions experimental investigation on spinodal decomposition are overviewed for the last five decades. Also, future developments in spinodal decomposition for modulated alloys are forecasted and criticized in an outlook.

Keywords: multi-materials; castings; microstructure

1. INTRODUCTION

Spinodal decomposition is a clustering reaction and as a result of this reaction homogeneous, supersaturated, solid or liquid solution splits suddenly into two phases. The twin phases produced by the spinodal decomposition of a supersaturated solid solution differ in composition from the parent phase, but have basically the same crystal structure. The physical implication of the spinodal is that it is the border between the unstable and metastable parts of a two phase region. This can be established by formative the sign of the alteration in free energy for a little composition fluctuation.

decompose into two phases along two dissimilar reaction paths [1].

The phase transformation that can produce a spinodal reaction object is decomposition within a stable or metastable miscibility gap (Fig. 1). If a solid solution of composition C_0 is solution treated in the single-phase field at a temperature T_0 , then aged at an intermediate temperature T_A (or T_A'), the single-phase alloy subjects to separate into a two-phase mixture. At the temperature T_A , the compositions of the twin phases α_1 , and α_2 under equilibrium conditions are C_1 and C_2 , correspondingly. Nevertheless, the supersaturated solid solution may

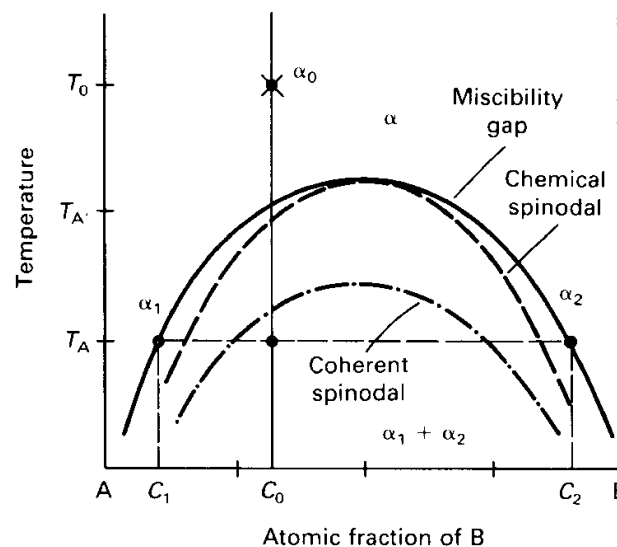


Figure 1 – Schematic showing miscibility gap in the solid state and spinodal lines (chemical and coherent) [1]

At small undercoolings or low supersaturations (TA'), the solution is metastable; exterior of a second phase engages comparatively large restricted composition changes. This is the classical nucleation procedure, giving rise to "critical nuclei," which can expand suddenly. As the particles of the newest phase expand by diffusion, the matrix composition regulates in the direction of equilibrium. At large supersaturations (TA), the solution is unstable, and the two-phase mixture gradually emerges by the enduring enlargement of mainly small amplitude changes (Fig. 2). The rate of reaction is directed by the rate of atomic migration and

the diffusion distances concerned, which depend upon the scale of decomposition (undercooling). Therefore, spinodal structures relate to phase mixtures that attain from a kinetic procedure leading the early stages of phase separation. The "spinodal line" revealed in Fig. 1 is not a phase boundary but a separation representing a difference in thermodynamic stability [1, 2].

In the current work, the advantages of spinodal decomposition, experimental investigations on modulated (spinodal) Cu-based alloys, and future developments in spinodal decomposition are reviewed.

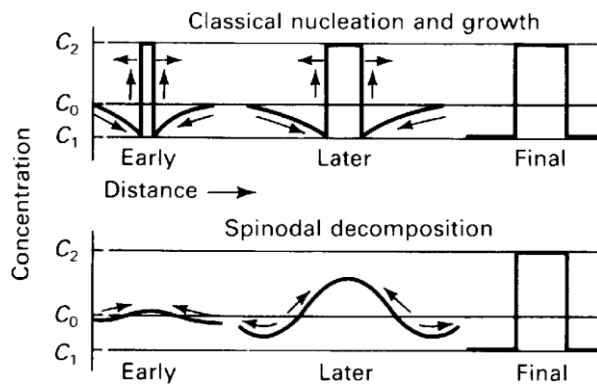


Figure 2 – Schematic illustrating two sequences for the formation of a two-phase mixture by diffusion processes: nucleation and growth and spinodal decomposition [2]

2. ADVANTAGES OF SPINODAL DECOMPOSITION

Precipitation hardening, dispersion strengthening and spinodal decomposition methods provide almost the identical strength values. Conversely there are definite advantageous of using spinodal decomposition process as against the precipitation hardening method. These are abridged here [3]:

- a) The decomposition-product phases have the identical structure but are dissimilar in composition, while in the case of precipitation the precipitate phase has a unlike structure and composition from that of the matrix. The principal advantage of the products having the identical structure is that microstructure is consistent. Consequently there are no local anodes and cathodes (eg grain boundary precipitates) to deteriorate corrosion resistance.
- b) In spinodal decomposition, there is no nucleation barrier and thus there is no need to offer activation energy. Therefore, hardening by spinodal decomposition is not predisposed by section thickness or quenching rate. Though, in the case of precipitation hardening, there is a nucleation barrier. Hence, if the radius of the precipitate nuclei is less than a critical value, the precipitate phases cannot be nucleated. As a result, the quenching rate and section size considerably influences enlarge of hardening in precipitation strengthening.

- c) The most significant advantage of spinodal hardening is that it is homogeneous throughout the section.
- d) Easy melting and casting are used to build the modulated (spinodal) alloy as against the P/M method or internal oxidation to be used for dispersion strengthening.
- e) At usual temperatures of application modulated (spinodal) alloys are not probable to over-age or recrystallize, while precipitation hardening alloys show a slow however exact over-ageing tendency.

3. EXPERIMENTAL RESEARCH

In this section, spinodal decomposition in copper, iron, titanium-based and other alloys will be summarized, as well as metallic glasses and polymers will be outlined in case of spinodal decomposition.

3.1 Spinodal decomposition in Cu-based alloys

A large number of investigators studied on Cu-Ni-Fe [4-5, 6, 7-11], Cu-Ni-Sn [3, 4, 12, 13-15, 16-17], Cu-Ni-Cr [18-19, 21,22, 23-25, 10, 26-28] and Cu based other alloys [29-31] using X-ray diffraction, transmission electron microscopy, electron diffraction, resistivity measurements and other techniques for example magnetic analysis and neutron diffraction.

a) Early stages of ageing

Various evidences of periodic decomposition products were detected in the electron micrographs of as quenched Cu-based specimens (Fig. 3). In the early stages of aging, the characteristics of spinodal decomposition examined via XRD and electron microscopy are: (i) sidebands on the XRD peaks, (ii) periodic structure in the electron micrographs (Fig. 4), (iii) first aging connected with steady wavelength (Fig. 5), (iv) lack of any power of grain boundaries or dislocations on the decomposition [19, 23].

The volume fractions of the phases at ageing temperature were determined from tie lines constructed via X-ray energy dispersive chemical analysis of the phases in combination with the analyzed alloy compositions. For instance, the volume fraction of the copper depleted phase was 0.12 for the Cu-30Ni-5Cr alloy (wt-%), and 0.46 for the Cu-45Ni-10Cr (wt-%) alloy at 800°C [24,25].

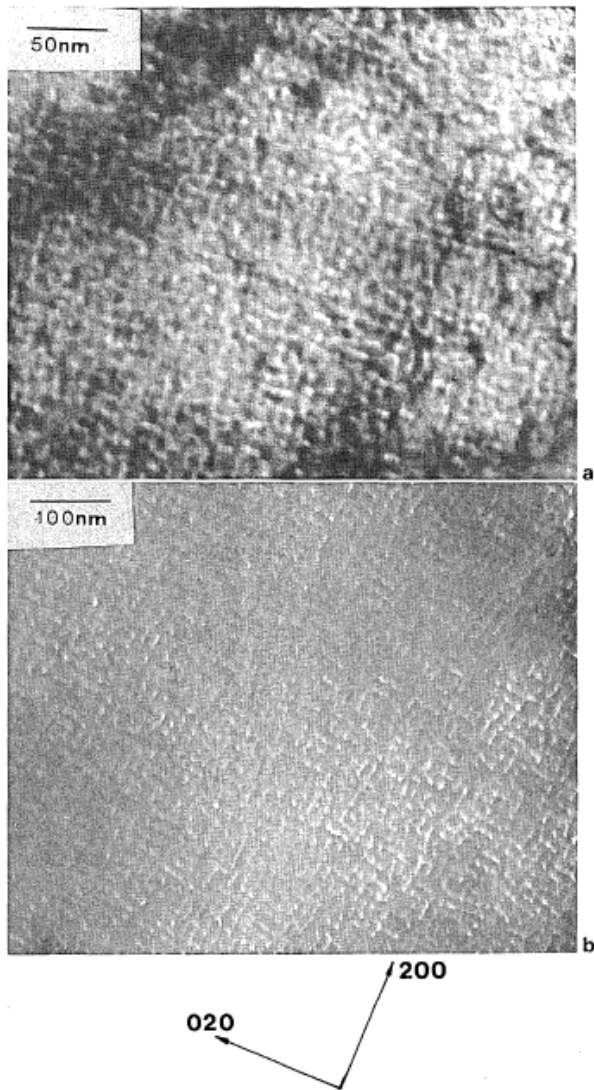


Figure 3 – Periodic structure in Cu-45Ni-10Cr alloy: $\lambda \approx 8$ nm (TEM), a) as quenched; b) aged 2 h at 300°C [19]

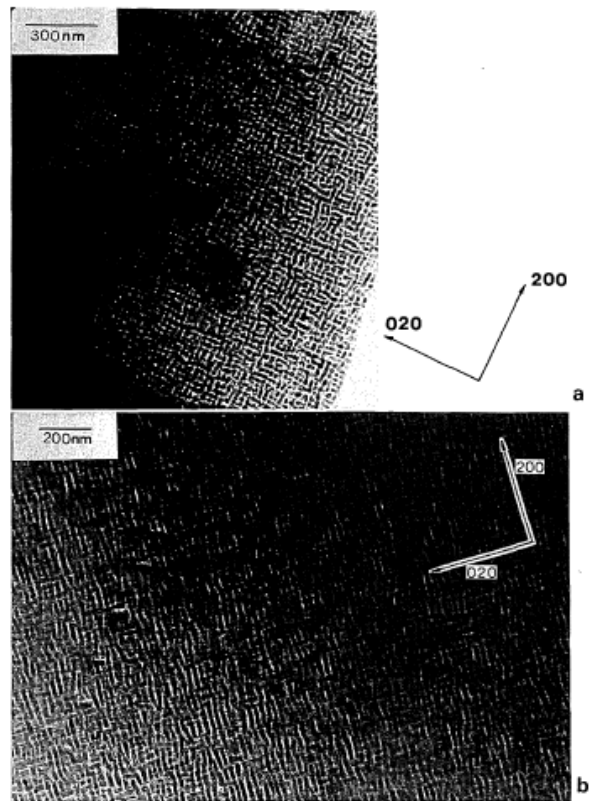


Figure 4 – Periodic structure in Cu-45Ni-10Cr alloy (TEM), a) aged 5 min, 800°C, $\lambda \approx 16$ nm; b) aged 1 day, 600°C, $\lambda \approx 17$ nm [19]

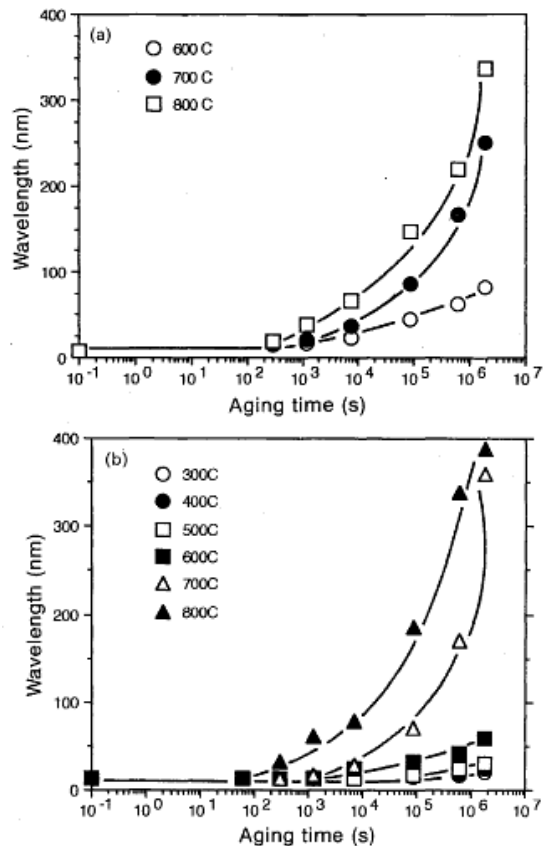


Figure 5 – Wavelength λ as function of aging time at various temperatures for a) Cu-30Ni-5Cr and b) Cu-45Ni-15Cr alloys [23]

In all Cu-based alloys for the period of the first stages of aging the particles were small, periodic, and cuboidal, and λ remained stable. Heterogeneous precipitation and precipitate free zones next to grain boundaries were not detected. Twin phases have all alloying elements and no pure phase was observed, in contrast to an earlier paper [32]. The particles clustered mutually and joined to shape periodic arrays of rods elongated in the $\langle 100 \rangle$ directions in both alloys as aging proceeded (Fig.4a). The precipitation characteristics detected are completely

reliable with spinodal decomposition. After extended aging times at high temperatures, interfacial dislocations developed at the particles (Figs. 6b and 6c) and grain boundary precipitation was detected (Fig. 6d). The previous is proof of loss of coherency and is connected with a reduction in hardness in the aging curves (Fig. 7). Following extended aging, the coarsened structure contained platelets. The morphological progress on aging can be summarized as cuboids \rightarrow rods \rightarrow platelets. These results are in harmony with prior investigation [33,34].

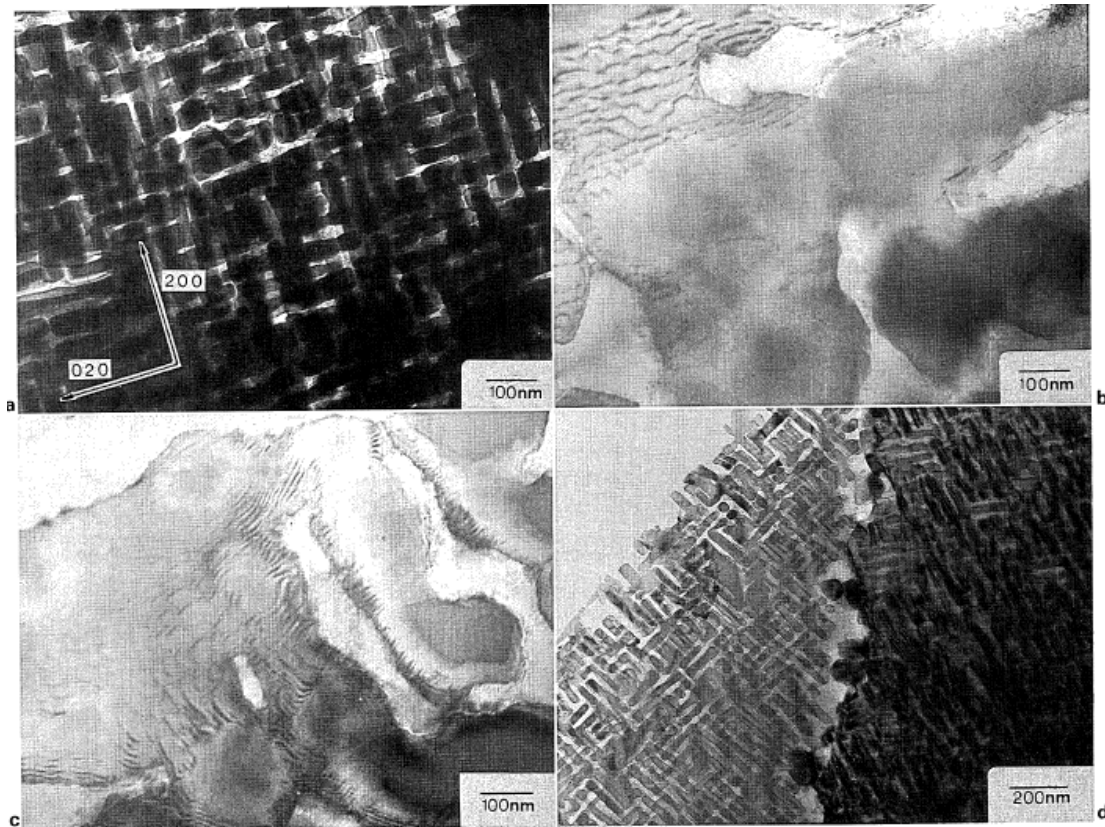


Figure 6 – Microstructure of Cu-45Ni-10Cr alloy following different aging treatments, a) feature spinodal decomposition structure after 3 weeks at 600°C, $\lambda \approx 40$ nm; b) interfacial dislocations after 1 week at 800°C, $\lambda \approx 330$ nm; c) coarse (platelet) structure after 1 week at 800°C, $\lambda \approx 330$ nm; d) grain boundary precipitation after 20 min at 800°C, $\lambda \approx 40$ nm [19]

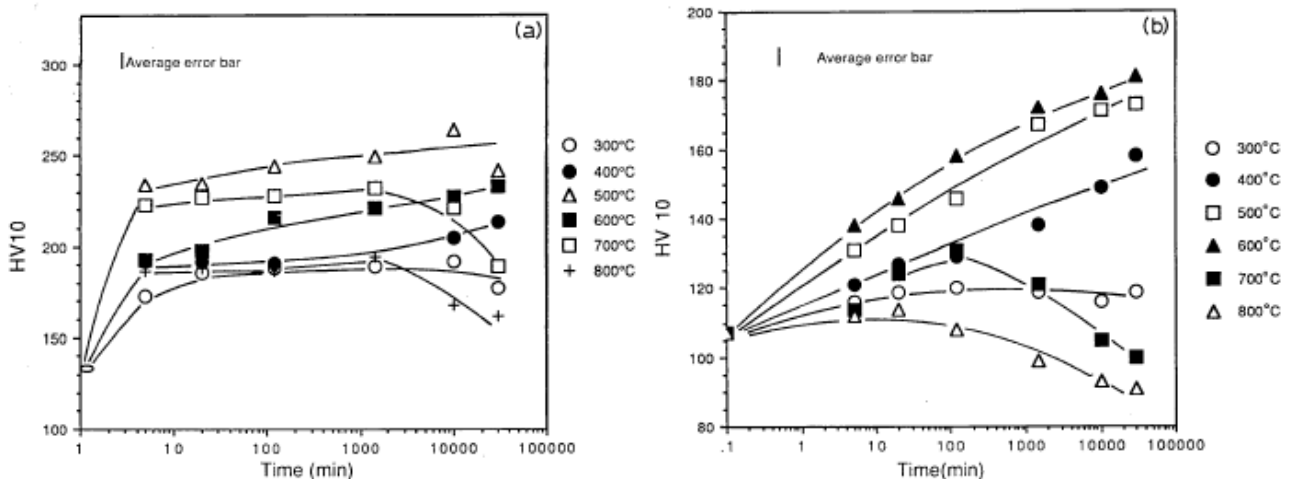


Figure 7 – Hardness (HV10) as function of aging time (min) for a) Cu-45Ni-10Cr and b) Cu-30Ni-2.5Cr alloy [19].

b) Later stages of ageing

In the Cu-based alloys, extended aging, particularly at higher temperatures resulted in the configuration of little spheroidal particles of a third phase (Fig. 8). Diffraction designated that these particles are bcc and, from EDX analysis, they were decided to be very rich in chromium. Their small dimension and the overlap of other phases in the thin foils excluded quantitative analysis although they seem to have some nickel and copper, opposing to an previous report of the formation of a 'pure chromium' phase [35]. In the Cu-45Ni-10Cr alloy, the microstructure of the decomposition products was not exaggerated by aging (Fig. 6d) and the cuboidal form stayed, even at the elevated aging temperatures. On the other hand, at 700 and 800°C, the cuboidal particle arrangement was uneven in distribution (e.g. Fig. 8) and sidebands were not observed on XRD patterns [19, 23]. By contrast, the cuboids firstly produced in the Cu-45Ni-10Cr alloy altered to rods aligned along (100) (Fig. 6a) and lastly to plates (Fig. 7b). This morphological change progression is in conformity with earlier reports [36, 33]. Loss of coherency and, progress of interfacial dislocation networks was also detected (Figs. 6b, 6c).

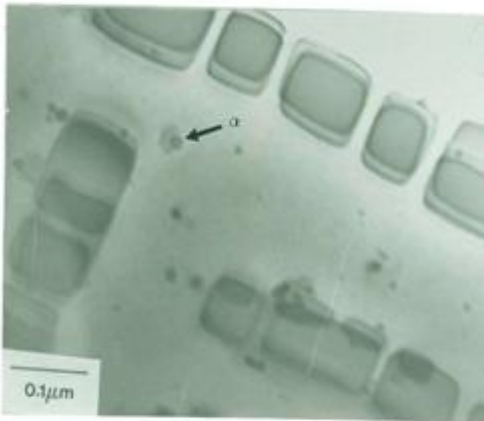


Figure 8– Electron micrographs of aged Cu-30Ni-5Cr alloy during the early stages of ageing. Structure lacks periodicity in all but 1 direction after 1 day ageing at 800°C, $\lambda \approx 1600 \text{ \AA}$ [23]

Coarsening of the decomposition products was chased by measurement of the wavelength of the periodic structure as a function of aging temperature and time. The wavelength of decomposition was deliberated either directly from the enlarged micrographs (large λ) or by calculation of the sidebands on the XRD patterns via the Daniel-Lipson equation: the 200 peak was utilized for measurement since it gave the most excellent arrangement of intensity and sideband resolution. The pertinent coarsening equation is [32, 37, 38].

$$\lambda_t^n - \lambda_0^n = k(t - t_0) \quad (4)$$

where λ_t is the wavelength of composition modulation at time t , λ_0 is the first constant wavelength, k and n are constants, and t_0 is the time at which coarsening begins.

The progress of wavelength with time and temperature for both alloys can be observed in Fig. 5. At inferior temperatures (300-500°C), measurement of the wavelengths was not easy, therefore these values are not incorporated in Fig. 5a. Also, in Cu-45Ni-15Cr alloy, wavelengths at 300 and 400°C were very comparable and consequently merely the values at 400°C are illustrated in Fig.5b. The coarsening performance of the Cu-45Ni-15Cr alloy determined from $\log \lambda - \log(t - t_0)$ graphs (Fig. 5b) is in conformity with equation (2) while the exponent $n = 3$. For other values of n the fit to the data is less good. Nevertheless, the Cu-30Ni-5Cr alloy did not follow this coarsening law particularly at lower temperatures (600°C) [24,25].

It has been revealed above that for the Cu-45Ni-15Cr alloy $\lambda \propto (t - t_0)^{1/3}$. Conversely, this does not directly designate the association between wavelength and particle size. The relationship between mean particle size p and wavelength λ for mutually alloys is exposed in Fig. 9. The slopes of the curves give the relationship between p and λ as $p \approx 0,580\lambda$ for the 5%Cr alloy and $p \approx 0.631\lambda$ for the 15%Cr alloy [19,23]. These results are in conformity with preceding investigation [39]. From graphs of time required for p to reach a fixed value (represented by $\ln(t - t_0)$) versus the inverse of temperature (Fig. 10), the subsequent estimated values of the activation energy for coarsening Q were obtained: 218 and 261 kJ mol^{-1} for the 5 and 15%Cr alloys correspondingly.

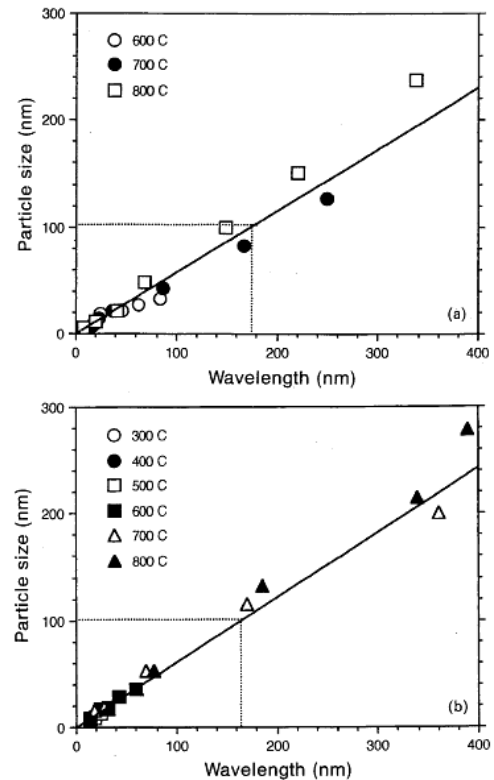


Figure 9 – Particle size as function of wavelength λ for various aging temperatures in a) Cu-30Ni-5Cr and b) Cu-45Ni-15Cr alloys [23]

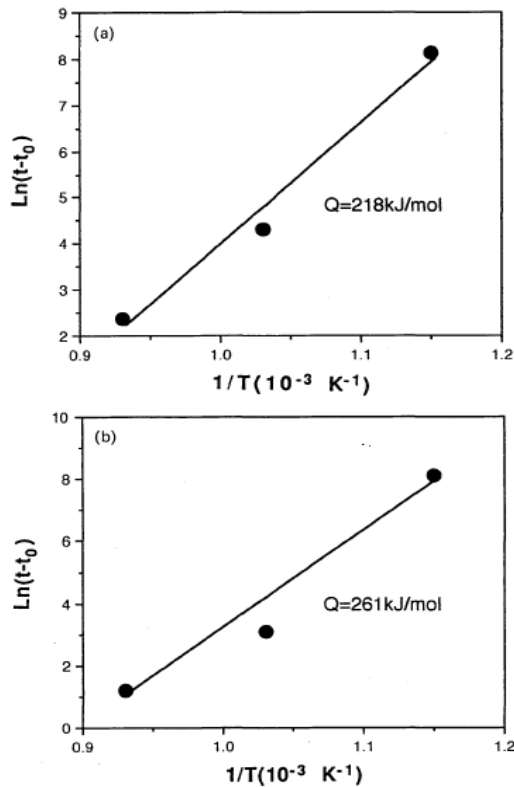


Figure 10 – Effect of temperature on time required to achieve particle size of a) 46 nm in Cu-30Ni-5Cr alloy and, b) 44 nm in Cu-45Ni-15Cr alloy [23]

Merely following extended aging times did precipitate free or denuded zones build up. This microstructural heterogeneity seems to derive chiefly from a comparatively fast loss of coherency of precipitate particles at grain boundaries and the special growth or coarsening of these particles at the expense of the coherent particles within the grains, as detected in earlier study [36].

The coarse precipitate structure and denuded area in the vicinity of the grain boundaries activate the migration of certain boundaries and a 'discontinuous coarsening' reaction finally involves the relocation of high angle grain boundaries and boundary diffusion, akin to cellular or discontinuous precipitation [36] (Fig. 12).

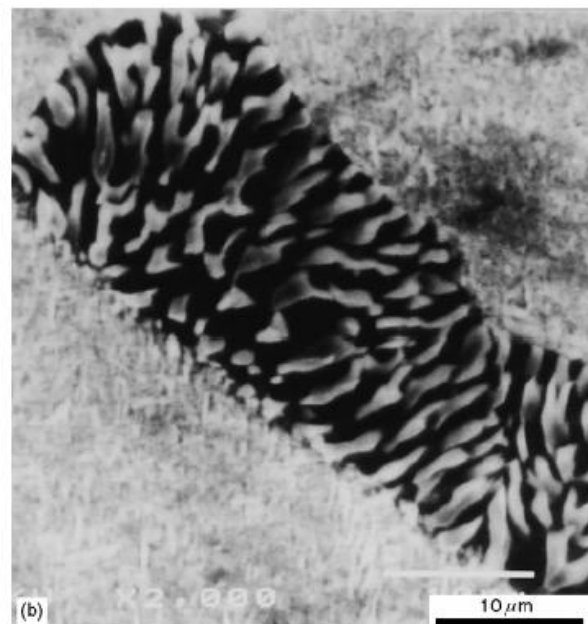
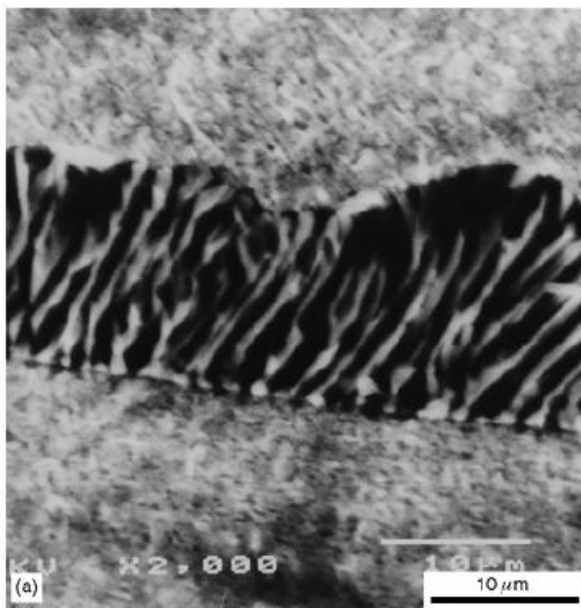


Figure 11– Scanning electron micrographs of different Cu-30Ni-5Cr alloys showing is continuous precipitation (2 h deep etched with Rosenthal's etchant). a) Discontinuous or cellular precipitation in C alloy after 3 weeks ageing at 700°C. b) Same as (a) but illustrating flake type structure (akin to tree branches) in the same alloy [40]

c) Age hardening

The hardness alteration was measured on specimens isothermally aged for diverse times in the temperature range 300-800°C and is exposed in Fig. 7. A very fast early rate of hardening can be observed in some Cu-based alloys (eg Cu-45Ni-10Cr in Fig. 7a), with an area showing a lower rate of hardening at longer times for all temperatures. The alteration from cuboids to rods was finished following very little aging times (~5 min) at

800°C, and the change from rods to platelets in 1 day, with a loss of coherency (Fig.6c). For temperatures in the range 300-600°C, hardness amplified continuously up to 10^5 min and a loss of coherency was not detected. Through this time, the precipitates distorted from cuboids to rods. At 700°C, the hardness curve illustrated a peak at ~100 min: coherency was preserved up to 1000 min at this temperature and subsequently loss of coherency started. Coarsening of the structure under these

conditions can be observed in Fig. 12. At this temperature (700°C) the morphological alteration progression can be printed as cuboids → rods within 1 day aging, and then rods → platelets.

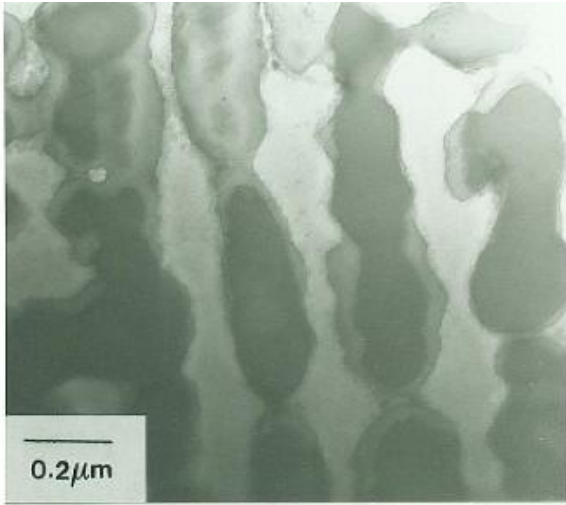


Figure 12 – Electron micrograph of Cu-45Ni-10Cr alloy during the later stages of ageing, showing more coarsened periodic structure (1 week aged at 800°C, $\lambda \approx 3300\text{Å}$) [19]

4. FUTURE DEVELOPMENTS

Spinodal alloys are compositionally neutral systems that show impetuous solute clustering on a nanoscale. This alteration occurs without a crystal structure change. Albeit spinodal decomposition was predicted by thermodynamics near the go round of the twentieth century, for a number of years, it was monitored only in ceramic systems. However, in the 1960s, with the beginning of TEM technology, spinodal decomposition was inspected in a number of alloy systems, counting the copper, iron and titanium alloys.

Right now, different spinodal alloys are intended for high strength applications, electrical and magnet alloys in addition to high speed cutting tools, for example cermets. For instance, today's copper–nickel–tin spinodal alloys are high performance wear resistant materials that function under arrangement of harsh load and speed. The outstanding characteristics of spinodal copper alloys have led to applications in large jet aircraft, highly difficult off-road equipment and heavy duty mechanical systems such as oilfield apparatus, which engages the extra advantage of corrosion resistance.

Alternatively, spinodal decomposition has been typically useful in the fabrication of stable magnet materials, because the morphologies favor high coercivities. The structure can be optimized by thermomechanical processing and magnetic aging. Permanent phase separation or spinodal decomposition appears to be important in the classic Alnicos and copper-nickel-iron

alloys, as well as in the recently developed iron-chromium-cobalt materials.

Spinodal decomposition is usually utilized in manufacturing and their application area is revealed as follows:

1. In electrical, automobile and building industries.
2. In ordnance and also in biological and agricultural areas.
3. Condenser tubes and seawater piping.
4. Manufacture of bullet envelopes.
5. Coinage making.
6. Electrical resistance and thermocouples.
7. Turbine blades production.
8. Used to produce heat exchanger tubes.

In spite of the obtainable papers considered so far, the following issues still need to be investigated concerning spinodal decomposition:

- (1) Novel alloying elements (such as high temperature elements ie Ti, Zr, Nb etc) can be added in order to delay the coarsening stage in the extended time ageing.
- (2) Supplementary corrosion tests can be performed on spinodal alloys after short and long period ageing time.
- (3) Wavelength-hardness connection can be compared to re-establish a universal hardening law for early and later ageing stages.
- (4) Discontinuous (cellular) precipitation worsens good mechanical properties of spinodal alloys. Thus, to hold back or restrain the cellular precipitation in all alloys, trace elements (e.g. Al, Si, Mg) should be added to the copper-nickel-chromium ternary system.
- (5) The bulk metallic glasses (BMG) have concerned great attention due to their exclusive properties and potential applications. Moreover many good properties, such as ultrahigh strength, good magnetic property, good anticorrosion property, and so on, they are also much cheaper in comparison to other BMGs.
- (6) The potential applications of polymer solutions in dense or supercritical fluids and mixtures will prolong to be strong in arrangement of particles, porous materials, blends and fiber. There is a clear prominence on developments of materials with nanoscale features particularly for the biopharmaceutical or biomedical applications.

The heat treatment for modulated (spinodal) alloys was chiefly utilized to develop the mechanical properties of metallic alloys such as iron and copper ones. More lately the treatment has moved away from simple heat treatment and is utilized to create in marine applications such as the hulls of small ships, the cladding of rudders of large ocean going ships, sheathing in the splash zone of fixed offshore structures, desalination plant and associated pipe-work pumps, valves, instrumentation and control systems. Potential developments of the

process might produce advanced structures for the nuclear and aerospace industry.

5. CONCLUSIONS

Spinodal decomposition is a system by which a solution of two or more components can divide into separate phases with clearly dissimilar chemical compositions and physical properties. This system differs from classical nucleation in that phase separation owing to spinodal decomposition is much more delicate, and happens homogeneously throughout the material. The following conclusions can be drawn from the current investigation:

a. The spinodal reaction is a spontaneous unmixing or diffusional clustering dissimilar from classical nucleation and growth in metastable solutions. This different kinetic performance was explained earlier by Gibbs in his treatment of the thermodynamic stability of supersaturated phases.

b. Precipitation hardening, dispersion strengthening and spinodal decomposition methods provide roughly the

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same strength values. However there are compact advantageous of via spinodal decomposition method, such as homogeneous microstructure, no influencing by section thickness and uniform all over the section, as against the precipitation hardening method.

c. Various alloys such as Cu, Fe and Ti-based, metallic glasses and polymers rendering to spinodal decompositions. In the early stages of ageing, these alloys demonstrate (i) sidebands on the XRD peaks, (ii) periodic structure in the electron micrographs, and (iii) early aging associated with steady wavelength. In the later extended ageing times, coarsening and precipitate free or denuded zones expand.

6. ACKNOWLEDGEMENTS

Particular thanks are included to the Sakarya University and the International University of Sarajevo for financial support.

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