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By

Rumyana Lazarova
and Lubomir A. Anestiev

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PART I:
PRODUCTION AND INVESTIGATION OF SURFACE AND BULK NANOCOMPOSITES

RUMYANA LAZAROVA

I dedicate my research work to the Metropolitan of Triaditsa
– His Eminence Photiy and my cleric -
His Reverence Stefan (Andonov)
as an expression of gratitude for the care,
they make for my soul.
Prof., Ph.D. Rumyana Lazarova

PREFACE TO PART I

The terms “metal-matrix composite” and “metal-matrix nanocomposite” are defined in the general introduction to this part. Laboratory methods for obtaining bulk and surface nanocomposites with an aluminum matrix are considered. The possibilities for industrial production of bulk aluminum nanocomposites are also briefly clarified. The application of aluminum nanocomposites, which comes down mainly to the replacement of aluminum microcomposites by them, is discussed.

The second chapter is devoted to surface nanocomposites obtained by incorporating nanoparticles in a substrate of aluminum and piston alloy using electron beam treatment. The improvement of the surface’s properties is due to the presence of hard nanoparticles, which take in the external load and prevent the dislocation movement in the matrix.

The third chapter describes a method for the preparation of bulk nanocomposites with an aluminum matrix consisting of cold isostatic pressing and hot extrusion of aluminum powders containing different amounts of nanoparticles. It is found out that the main strengthening mechanism in the investigated nanocomposites is the Orowan mechanism.

The fourth chapter is devoted to the formation of the nucleus and microstructure in an aluminum nanocomposite reinforced with TiCN nanoparticles. The dependence of the rate of nucleation in aluminum on the size of nanoparticles with a cubic shape was determined. The process of growth and modification of dendrites and silicon crystals in aluminum alloy is described. The process of microstructure formation in a sample of AlSi12Cu2NiMg alloy subjected to electron beam treatment was simulated using the MAGMASOFT® software. Conclusions are made in the fifth chapter.

The first part of the book is intended for several groups of readers: researchers working on the development and research of nanocomposites based on aluminum and metal in general, university professors of metal science and materials science, students and Ph.D. students in materials science, metal science, nanosciences and nanotechnologies, manufacturers of metal-based nanocomposites and all readers who are interested in surface and bulk metal-based nanocomposites.

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ABOUT THE AUTHOR

Dr. Rumyana L. Lazarova is a Professor of Materials Science at the Institute of Metal Science, Equipment and Technologies with Hydro- and Aerodynamics Centre (IMSETHAC), at the Bulgarian Academy of Sciences (BAS). She received her master's degree in engineering from the Technical University – Sofia, Bulgaria, and her Ph.D. degree from the Bulgarian Academy of Sciences. In 1995 Dr. R. Lazarova joined the department of Materials testing and Analysis at the Institute of Metal Science and Technology, BAS (the predecessor of IMSETHAC). During her employment at that time, Dr. R. Lazarova worked on several projects in materials science. From 1999 to 2000 she was a scholarship holder at UCL Louvain-la-Neuve, Belgium. Her research interests are currently focused on graphene-reinforced aluminum nanocomposites.

She is the author of over 110 scientific publications.

CHAPTER I-1

GENERAL INTRODUCTION

RUMYANA LAZAROVA

1.1 Aluminum matrix nanocomposite: definition and general characterization

The concept of composite material is sufficiently known and clarified in modern material science [1, 2, and 3]. It is assumed that “any material consisting of two or more components” with “distinct boundaries” between them and “different properties” is a composite material [3]. With greater precision, we could define it as follows:

- Composite material contains two or more chemically separate phases;
- The quantity, form, and distribution of the constituents are controlled in a predetermined manner during the composite manufacturing;
- The composite has unique properties, which are determined by the quantity, shape, and distribution of phases through the principles of mechanics.

The composite usually consists of base or matrix material which as a rule is homogenous and isotropic. Its properties and behavior could be described by the mechanics of conventional materials. The properties and the behavior of the matrix are improved by filling it with particles, fibers, or plates of another material which is named filler or reinforcement.

Metal matrix composites (MMCs) are divided into three groups: *particle reinforced*, *short fiber-reinforced,* and *continuous fiber-reinforced MMCs*. Particles contained in the first category MMCs are ranging from 10 nm up to 500 µm. **MMCs with a uniform dispersion of particles in the range of 10 nm to 1 µm are named — Metal Matrix Nanocomposites (MMNCs)**. On this basis, **nanocomposites can be defined as composites in which the filler has dimensions in the nanometric range (1 nm = 10^{-9} m)** [4]. This category includes nanocomposites with ultrafine particles. These are particles that are less than 100 nm in diameter. MMNCs possess more outstanding properties than MMCs which overcome the disadvantages of MMCs for example poor ductility, low fracture toughness, and machinability.

The classification of nanomaterials can be made based on numerous characteristics. The main feature is the nature of the constituents - organic or inorganic. The second characteristic could be the dimensionality of constituents – one, two, or three-dimensional.

We investigate the case in which one constituent is a three-dimensional matrix and the other one represents nano-sized particles distributed in the matrix. The chemical nature of the matrix is metallic and the nanoparticles play the role of reinforcement - ceramic. That means metal matrix nanocomposite (MMNC) is a combination of metal matrix and nano-sized reinforcement (usually ceramics) [5]. Generally, MMNCs are metals reinforced with other metal, ceramic or organic compounds with nano-dimensions.

Metal matrix nanocomposites (MMNCs) consist of a ductile metal or alloy matrix in which nano-sized reinforcement material is incorporated. These materials combine metal and ceramic features - ductility and toughness with high strength and hardness. Because of the nano-metric size of the reinforcement phase, the interaction of particles with dislocations becomes of significant importance for strengthening and, when added to other strengthening effects typically found in conventional metal matrix composites (MMCs), leads to a significant improvement of mechanical properties [6-10].

The references on a micrometric particle in MMCs show that although micrometric dispersion is easier to achieve, these composites are less effective in strengthening compared to MMNCs. For example, the tensile strength of 1 vol. % Si_3N_4 (10nm) – aluminum MMNC is comparable to that of 15 vol. % SiCp (3.5μm) – aluminum MMC, while the yield stress of the MMNC is significantly higher than that of the micrometric particle dispersed MMC [11]. Moreover, particles larger than 1.5μm act as micro concentrators and cause cleavage between them and the matrix. Particles with dimensions between 200 and 1500 nm cause the formation of cavities and pits due to their poor interphase cohesion. But particles with a size less than 200 nm bond well with matrix and so that determine excellent mechanical properties of MMNCs [12]. We could conditionally assume that MMNCs have one-dimensional reinforcements because the particles have sizes below 200 nm, unlike MMCs in which the reinforcements are except particles with dimensions above 200 nm and fibers and plates.

Due to their nano-dimensionality, the particles that serve as reinforcements in MMNCs have special properties that need to be highlighted.

When the particle size is brought down to the nanometric length scale, surface energy is increased by three orders of magnitude [13]. This engenders instability in a gas-liquid-solid system, the Gibbs energy of which, is described by the following equation:

$\Delta G = (\mu G(T,P) - \mu L(T,P)) + \gamma_{LG}\,\Delta S_{LG} + \gamma_{SG}\,\Delta S_{SG} + \gamma_{SL}\,\Delta S_{SL},$

where T is the temperature, P the pressure in the liquid, μG and μL the chemical potentials of gas and the liquid, and ΔS is the change in interfacial areas and γ surface energies.

In this situation, agglomeration of nanoparticles, which provides a smaller interfacial area, is proving to be an energy-efficient phenomenon: several nano-particle clusters in one microagglomerate.

The dynamics of the relative motion of two nano-sized particles have been extensively studied [14]. Due to the complexity of the problem, the analysis is usually limited to two main mechanisms: Brownian diffusion and inter-particle forces (electrostatic and Van der Waals).

Brownian diffusion, which is identified with aggregation limit factor, ensures the continuous collision between particles. The magnitude of the displacement follows a Gaussian statistic distribution according to the relation:

$$\theta = \frac{-Ar}{12H^2}$$

where A is the Hamaker constant, which depends on the polarization properties of the molecules on the particle surface, r is the reduced particle radius, and H the inter-particle distance [14]. When the particle dimension is smaller than 1 μm, Van der Waals's forces dominate. Coulomb's force of repulsion competes with Van der Waals force of attraction. The Van der Waals attraction force overcomes the electrostatic repulsion for an inter-particle distance down to 1 nm. For smaller values, the Born repulsion of adjacent electron clouds dominates [15].

The essential for an effective load transfer from the matrix to the particle and for delaying the onset of particle-matrix decohesion is the good interface bonding between particles and matrix.

Thus, MMNCs are suitable for the production of materials with high strength in shear-compression processes and wear resistance. They show extraordinary potential for application in many areas, such as the aerospace and automotive industries and the development of structural materials [16].

The matrix of composites we study is aluminum. Aluminum and aluminum alloys are of interest as parent metals in MMNCs. Commonly used reinforcements are carbides, borides, nitrides, and oxides, i.e, SiC, TiC, TiCN, Al_2O_3, WC, TiB_2, and others. The effectiveness of the nanoparticles is expressed normally only between 0.5 and 5% addition by weight, but sometimes it reaches 10-15%.

The result of the addition of nanoparticles is a drastic improvement in properties such as mechanical strength, toughness, hardness, and wear resistance.

By surface aluminum nanocomposites we mean thin coatings or surface layers of nanocomposite on an aluminum substrate. Depending on whether the aluminum nanocomposite is bulk or surface, its purpose as a machine part in modern engineering could be different.

There are many applications in which only surface properties play an important role for example wear resistance. In this situation, only the surface layer needs to be reinforced by ceramic phases while the bulk of the component should retain the original composition and structure with higher toughness. There are several methods to fabricate particulate reinforced nanocomposites to create functional surfaces as well as many methods for the production of bulk metal nanocomposites. Below we have considered the possibilities for obtaining metal nanocomposites.

1.2 Methods for nanocomposites producing

1.2.1 Methods for bulk nanocomposites producing

There are two categories of metal matrix composites, respectively nanocomposites, depending on the manufacturing process: ex-situ and in-situ. Ex-situ composites are obtained when the reinforcement is externally added to the matrix. In-situ fabricated metal matrix composites are characterized by the production of reinforcements within the matrix during the fabrication process.

On the other side, the methods for metal matrix nanocomposites manufacturing could be classified depending upon the matrix state during the production process, which can be molten or solid [1, 17-20].

Solid-state processing

Solid-state processes for fabrication of bulk nanocomposites relate to powder metallurgy. This method provides better wetting of the nanoparticles and a better matrix-reinforcing particle bond. Furthermore, this processing route can incorporate a higher volume fraction of reinforcement.

- **High energy ball milling**

The raw materials are breaking up into nano-scale sizes and mixed then utilizing rotation and vibration of ball milling machine. The obtained product is a pure element, alloy, or nano-composite powder. For producing bulk nano-materials hot extrusion or hot isostatic pressing could be used then.

- **Powder metallurgy (powder blending and consolidation)**

Powder metallurgy (PM) is a manufacturing approach, in which many kinds of powder technology can be used [21]. The constituent processes of this route are: blending matrix and reinforcing powders; compacting the blend, usually by cold pressing; degassing the compacted product to remove water vapor and gases; consolidation of the slugs by one of the following methods: direct sintering, hot isostatic pressing, vacuum hot isostatic pressing, hot extrusion, and cold sintering.

Nano-sized particles are manufactured first and then mixed with metal matrix powders. The next processes are used for obtaining a bulk product [22]. Powder metallurgy is a very attractive technique as it employs lower temperatures and thus, ensures better control of interface kinetics.

Powders of aluminum or aluminum alloy are blended with the nano-reinforcement. Blended powders are then compacted into a ‘green form’, using uniaxial pressing or cold isostatic pressing. The green body thus obtained is approximately 80% dense. The green body is heated usually in an inert atmosphere to a desired processing temperature to obtain a sintered body [23]. The sintered body has a 100% theoretical density.

- **Severe plastic deformation**

Severe plastic deformation (SPD) is a method by which nano-structured materials are fabricated. Extremely large refinement of the microstructure can be achieved through it. The refinement is expressed by structure elements size reduced into sub-micron or even nano-scale. This is obtained by the very large strains imposed on the specimen at a temperature usually less than the temperature of recrystallization. High densities of crystal lattice defects (particularly dislocations) are created as a result and form nano-sized grains and subgrains.

The two main fabrication routes for SPD are severe plastic torsional straining (SPTS) under high pressure and equal channel angular pressing (ECAP).

- **Mechanical alloying**

The blending of the powders in the traditional PM occurs without material transfer between the mixed components. It is possible, however, to incorporate hard ceramic particles into the matrix powder particles through a solid-state bonding [24] which performs a mechanical alloying. This process could be determined also as cold welding between the different types of particles. Mechanical alloying could be defined as a solid-state powder processing technique consisting of repeated cold welding, fracturing, and rewelding of powder particles in a high-energy ball mill. Many types of aluminum-based nanocomposites were produced by this method for example Al–AlB_2, Al–Al_4C_3, Al–Al_xMo_y, Al–AlN, Al–Al_2O_3, Al–Al_3Ti, Al–BN, Al–C, Al–$CuAl_2$, Al–MgB_2, Al–SiC and Al–TiC [25].

- **Microwave and bi-directional hybrid microwave sintering**

These processes are based on the conversion of electromagnetic energy into thermal energy, which is rapidly and highly efficiently spreading. The heat is generated from within the powders and radiates outward due to the penetrative power of the microwaves. Higher temperatures exist at the core of sintered materials whereas the surfaces demonstrate lower temperatures. Because of that, the variation in microstructure along the thickness of the sample is observed which results in bad properties. This problem is resolved by using bi-directional hybrid microwave sintering, wherein microwave susceptors such as SiC particles/rods assist in the reduction of thermal gradient during sintering.

Semi-solid state processes

Semi-solid processes consist of shaping a partially solid mixture in which the solid fractions are between 20 and 60 % [20]. The advantages of these processes are low shrinkage and porosity, non-turbulent filling, and lower processing temperature. They can be divided into two main groups: thixo-processes and rheo-processes (compocasting). The thixo-processes use solid feedstock which is partially melted. The base material is obtained by partially solidifying a liquid melt under controlled conditions for inducing the formation of crystals in the slurry [20]. The thixo-processes need to be adapted and optimized for the production of metal matrix nanocomposites. The authors of [20] consider **thixoprocessing** as a key technology for the industrial scaling up of MMNCs production because significant effects on properties can be obtained at low volume fractions of particles. The prewetted feedstock, which can be prepared in several methods, allows handling nano-reinforcement on a smaller volume scale before diluting the master composite in a larger metal volume. Further research is needed for synthesizing the nanocomposites in an industrial process to utilize these promising materials in various applications. The rheocasting could be combined with squeeze casting for the production of aluminum nanocomposites reinforced with nanoparticles [26].

Liquid State Processes

Liquid state processing routes for MMNCs producing are relatively simple, cost-effective, and suitable for industrial use. These routes are as follows:

- **Stirring techniques**

It is indisputable that the **stir casting process** is a suitable method for micron-size reinforcement composites producing, but for nano-sized reinforcement's composites fabrication, it poses an especially great difficulty – the dispersion and uniform distribution of nanoparticles in industrial-scale melts due to the large surface-to-volume ratio [27].

The nanopowders are very often added to the liquid matrix and distributed in it by applying mechanical stirring through an impeller. Because of the poor wettability of nanoparticles within the molten metal and their tendency to agglomerate, it is usually difficult to obtain a homogeneous distribution of the nano-reinforcement. The difficulties in mixing and wetting nano-sized particles by metallic melts require particles pre-coating or cladding with metal-protector. The as-prepared molten alloy with the dispersed reinforcing nanoparticles may be used for sand casting or permanent mold casting for obtaining a bulk nanocomposite.

- **Droplet consolidation techniques**

Droplet consolidation could be defined as a "rapid" quenching from the liquid state. The cooling rate, which is usually greater than 1 mm s^{-1} characterizes these techniques.

- **Ultrasonic assisted casting**

The characteristic feature of this method is the melt treating with ultrasonic waves to disperse homogenously the reinforcement. This method is used for the production of aluminum-based nanocomposites reinforced with different types of nanoparticles. The ultrasonic-assisted casting is considered as a method providing better matrix–particle bonding, braking nano-particle clusters, and removing impurities from the surfaceof the particles, which assures good microstructural characteristics [20]. However, the ultrasonic casting is difficult to be scaled up to the industrial level as the volumes of castings are limited to the dimension of the ultrasonic probe and power of the ultrasonic source. These limitations could be overcome if ultrasonic flow processing could be adopted. This concept was used in developing a scaled-up ultrasonic processing system, aimed to produce MMNCs at an industrial scale [20].

- **Infiltration technique**

The infiltration process consists of preparing a porous "preform" of the reinforcement and infiltration its pores with the molten metal. The flow of molten metal is driven only by the forces of capillarity i.e. pressure is needed to overcome the resisting forces due to drag and capillary. The required pressure is a function of the friction effects due to the viscosity of the molten matrix. Wetting the ceramic preform by the liquid alloy depends on different factors like alloy composition, ceramic preform material and surface morphology, temperature, and time. The pressure/vacuum infiltration method is already a widely used method for composites with micron-size reinforcements producing. However, its use for large-scale MMNCs production depends on the preparation of nano reinforcement preform, and the parameters of the infiltration process. Due to these reasons, the large-scale production of MMNCs by pressure/vacuum infiltration requires a highly standardized process procedure with accurate control of process parameters by which an eventual preform breakage, increased porosity and/or phase segregation could be avoided.

The infiltration technique could be applied in a pressureless configuration. In this case, a spontaneous infiltration of the ceramic preform acts without the aid of externally applied pressure, nor vacuum. B. Xiong et al. produced aluminum alloy-based nanocomposites reinforced with SiC particles by combining ball-milling and cold pressing to make the preform and then used a pressureless infiltration [28 and 29].

The infiltration techniques are characterized by long infiltration times and preform costs.

- **The disintegrated melt deposition technique** has some advantages in respect to MMNCs production - fine grain size, uniform nanoparticle distribution, minimum porosity, and superior mechanical properties [30]. Since the microstructural and mechanical properties depend on parameters of this process such as the disintegrating gas velocity, distance between the melt exit stream and the substrate, etc. which have to be varied according to the dimension of the final product, the DMD process is more suitable to produce MMNC ingots to be used as precursors for making wrought products. Also, due to its set-up (see Sect. 6.2), the DMD process is difficult to be automated and to be used for continuous casting operations [30].
- **High pressure die casting**

This method consists of feeling the die cavity by the molten metal under pressure. Both filling speeds and solidification rates are relatively high. The technique is suitable to obtain detailed components. It could be used for producing nanocomposites and especially aluminum nanocomposites but there are feu works as examples for that [31 and 32].

Homogenous distribution of the nano-sized reinforcement phase is difficult to achieve using liquid-processing methods because of the difference in densities between the two components of the composite. The poor wettability of the reinforcement by the molten metal makes mixing very difficult leading to a heterogeneous structure that affects the properties of the composite.

Combined or hybrid methods

Nanocomposites can also be produced through a combination of the based processes discussed above. Powder-metallurgy techniques are usually applied to producing the nanocomposite which further is introduced and imported into the liquid or semi-liquid metal.

- **Friction stir processing**

Friction stir processing is based on friction stir welding which is a technique for surface metal matrix production. However, in recent years, it is used as a route to incorporate nanoparticles into the metal matrix to fabricate bulk nanocomposites. A rotating tool with a shoulder and a pin are plunged into the surface of the workpiece (the desired base matrix) with grooves filled with the desired volume fraction of nanoparticles [20].

- **Accumulative roll bonding**

In essence, this process presents a severe plastic deformation allowing producing nanostructured and ultrafine-grained materials. It consists of roll bonding stacked sheets sprinkled with nanoparticles, then cutting the sheets and rolling them again after stacking the pieces over each other.

As we have seen, when considering the individual methods for preparing metal-based nanocomposites, they are currently obtained mainly in the laboratory. Their production on an industrial scale is still a significant challenge for scientists and engineers [33]. This is due to their recent development. For this reason, it is good to look briefly at the problems of each method of producing aluminum matrix nanocomposites, its use in industrial conditions, and the fabrication of an industrial product.

1.2.2 Methods for surface nanocomposites producing

When the surface of the component is required to be of higher strength, hardness, and wear resistance, and the core to maintain its plasticity and toughness, it is appropriate to apply a superficial nanocomposite. The modified or applied surface layer is named surface metal matrix nanocomposites (SMMNCs).

Fabrication of SMMCs is usually performed using surface treatment techniques such as high energy laser beam, plasma spraying, cast sinter, and electron beam irradiation which have been developed over the last decades [34].

The methods for surface nanocomposites fabricating could be classified as follows [35]:

- **Cold spray method**

Cold spraying allows the fabrication of coatings at lower temperatures than the melting points of the used materials. Thus this method avoids the deterioration phenomenon of the materials such as oxidation and decomposition as well as phase transition during the process. The obtained coatings have low porosity (<1%) and low oxygen concentration. In addition, the coatings have high strength (>280MPa) and strong adhesion (>70MPa). This method is used to produce nanocomposite coatings with metallic or alloy matrices. The nanofillers, in this case, are nitride, carbide, boride, diamond, CNT, etc. For the production of nanocomposite powders, mechanical alloying is used with metallic matrix powders and other nanoparticles.

- **CVD (chemical vapor deposition) method**

This method is usually used for fabricating coatings of inorganic matrices and inorganic nanofillers. The aerosol-assisted CVD method can be used for improving the quality of the coating.

- **PVD (physical vapor deposition) method**

This method serves to produce nanocomposite coatings of inorganic matrices and inorganic nanoparticles. In this case, the PVD method includes the processes: laser ablation, thermal evaporation, ion beam deposition, ion implantation, laser-assisted deposition, and atom beam co-sputtering technique. For nanocomposite coating with organic matrix, aerosol-assisted plasma deposition is used [36].

- **Thermal spray method**

This method is often used to make nanocomposite coatings with a matrix of metal or alloy. The spray material is a nanosized alloy powder (formed by ball milling) and dispersed in a suspension solution using suspensions to conduct plasma thermal spraying.

- **Welding techniques**

Methods for the introduction of nanoparticles during manual arc welding, electron-beam welding, MIG/MAG, and TIG welding are presented in [37 and 38]. The manners for metal microstructure modifying are in principle the same as the manners for surface nanocomposites manufacturing.

Electron beam irradiation is an advanced technique for surface modification as laser beam irradiation, ion implantation, etc. [39]. We use this technique for surface nanocomposite production. The surface aluminum matrix nanocomposites we have produced and investigated have been obtained using coating the substrate surface with a mixture containing nanoparticles and the following electron beam scanning treatment incorporating the reinforcement in the aluminum matrix.

1.3 Application of aluminum matrix nanocomposites

The wide use of aluminum matrix composites is determined by their properties which cannot be achieved by any monolithic material. It is logical to assume that aluminum matrix nanocomposites would find similar applications as those of aluminum composites. Here we would like to recall some industrial applications of aluminum composites:

1.3.1 Application in the automotive engineering industry

Aluminum matrix composites have a great application in the automotive engineering industry for the production of valves, piston rods, pistons, piston pins; covers: cylinder heads, crankshaft main bearings; engine blocks: part strengthen cylinder blocks; brake rotors, calipers, liners, propeller shafts, connecting rods, piston rings, rear brake drums, driveshaft, engine cradles, multichip electronic module. It is due to their properties as good strength to weight ratio, high specific strength, low coefficient thermal expansion, and high thermal resistance, good damping capacities, superior wear resistance, high specific stiffness, and satisfactory levels of corrosion resistance exceptional microstructure stability at high temperatures [40].

1.3.2 Aerospace and aircraft application

The aluminum matrix composites are applied for producing: wings and supporting structures in airlines, fuselage, military aircraft and cargo, aircraft electrical ac doors, heat sinks and missile fins [19].

1.3.3 Defense industry application

Passivated aluminum composites are used for enhancing desirable characteristics for propellant applications [41].

1.3.4 Building and construction materials

Strength and stiff-propeller shafts and brake disc are fabricated.

Others as: rail transport, marine transport, electrical transmission, sports and recreation, etc. [42].

As nanoparticle reinforced MMCs show higher mechanical properties than micro-particles reinforced composites without decreasing thermal and electrical conductivity, they are ideal candidates for substituting conventional MMCs or analogous monolithic alloys in structural and electrical applications. Lightweight metal matrix nanocomposites with their high wear resistance, and thermal conductivity, could be employed in automotive industries as brake system components, as well as piston liners owning to their high wear resistance, good thermal conductivity, and low coefficient of thermal expansion or as valves owning to their high creep resistance and resistance to sliding wear [43]. Furthermore, because of the high specific strength and elastic modulus of lightweight metal matrix nanocomposites, they could find good use in the sports industry, like rackets, bicycle frames, and other components. Another field of application is in electronic devices (heat sinks and solders thanks to their thermal properties or as antennas thanks to their electrical properties and stiffness) [43].

Thus, most of the products of conventional composites can be substituted by MMNCs. These are ventral fins for aircraft and exit guide vanes in the aerospace industry. Brake system components in the automotive industry, bumper beams, and connectors, inner hood, steering knuckle, crash support beams, wheels, etc. MMNCs are ideal candidates in thermal management and electronic packaging systems such as radiator panels and battery sleeves, power semiconductor packages, black box enclosures, and printed circuit board heat sinks [20].

The next few decades will reveal the widespread use of aluminum nanocomposites in many industries.

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CHAPTER I-2

SURFACE NANOCOMPOSITES ON ALUMINUM AND PISTON ALLOY SUBSTRATE

RUMYANA LAZAROVA

For the development of this chapter, we have used the following articles with the kind consent of the publishers Taylor&Francis and IOP Conference Series: 1. R. Lazarova, R. Dimitrova, Y. Murdjeva, St. Valkov, P. Petrov, *Layers obtained on aluminum by nanopowder deposition and subsequent electron beam scanning*, Materials and Manufacturing Processes, Taylor&Francis, 2018, ISSN:1532-2475, DOI:10.1080/10426914.2018.1453148, 1-5; 2. Stefan Valkov, Ruslan Bezdushnyi, Rumiana Lazarova, Rossitza Dimitrova, and Peter Petrov, *Surface modification of Al substrate with TiCN nanopowder by electron-beam treatment*, AIP Conference Proceedings 2075, 160017 (2019); https://doi.org/10.1063/1.5091344 ; 3. R Lazarova, S Valkov, V Dyakova, and P Petrov, *Layers obtained on TiCN aluminum nanocomposites by electron-beam treatment*, in IOP Conference Series: Materials Science and Engineering, International Conference on Novel Functional Materials IOP Conf. Series: Materials Science and Engineering 733 (2020) 012017 IOP Publishing, doi:10.1088/1757-899X/733/1/012017, 1-9 and 4. Mihail Kolev, Stefan Valkov, Rumyana Lazarova, Peter Petrov, Rossitza Dimitrova, and Vanya Dyakova, Tribological Properties of TiCN Nanosized Powder Reinforced Aluminum, Journal of Physics: Conf. Series, 992, IOP Publishing, 2019, 1492(1), 012066

2.1 Introduction

The aluminum and aluminum alloy nanocomposites are designed to have better mechanical and tribological properties than aluminum and corresponding aluminum alloys used in automotive and aircraft engineering. Because of that their microstructure and properties should be characterized and then compared with those of the aluminum and respective alloys.

We have elucidated the methods for surface nanocomposites producing in Chapter 1.2.2 of this book. Now we will demonstrate the results we obtained using surface treatment of aluminum and aluminum alloy with concentrated energy flows (or fluxes) as a method for manufacturing surface nanocomposites.

Surface treatment of metals and alloys with concentrated energy fluxes (CEF), such as laser beam and electron beams, has been intensively used over the last several decades. Their main advantage is the possibility of precise control of the technological conditions of processing, which in turn allows controlling the structure and properties of the processed materials. When the flow of accelerated electrons hits the surface to be treated, the kinetic energy of these particles is transformed into heat, which propagates into the volume of the sample [1-4]. The technological parameters can be selected so that the treatment will bring the modified surface to the liquid phase - formation of a liquid bath. In the preparation of composite materials based on aluminum and aluminum alloys with introduced nanoparticles, the nanoobjects are introduced into the liquid phase in powder form or pre-deposited on the surface as a coating. For their homogeneous distribution, the technological conditions of electron beam treatment (EBT), defined by the technological parameters, are of fundamental importance: accelerating voltage (U, kV); electron beam current (Ib, mA); the speed of movement of the sample during the EBT (V, mm / sec); scanning frequency (f, Hz).

It has been found that the distribution and homogenization of alloying elements in the case of introduction by electron beam treatment with the formation of a liquid bath are based on the intense convection of Marangoni generated by the high-temperature gradient in the liquid bath. The influence of technological conditions on the formation of aluminum nanocomposites was investigated, and it was determined that the speed of movement of the specimen was essential in their preparation. The high speed of movement of the sample results in a short lifetime of the liquid bath. In this case, the transfer of the elements in the liquid bath is extremely insufficient for their homogeneous distribution, which leads to the formation of highly inhomogeneous structures. At low sample movement rates during the EBP, the main mechanism for the transfer of elements in a liquid bath is based on the convection flows that exist due to the presence of a high-temperature gradient. The existence of the liquid bath is much larger, which leads to the formation of homogeneous composite structures. It has also been found that the application of high scanning electron beam frequencies leads to a longer lifetime of the liquid bath and significantly more suitable conditions for the introduction of the alloying elements as well as their homogeneous distribution. It has been proven that the technological parameters of the EBP have to be chosen so