Nano Ceramic Matrix Composite Development and Its Applications

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ABSTRACT

The nano ceramic matrix composite is used in variety of applications due to its unique physical properties and capability to perform better at elevated temperature. Research is going on worldwide to understand the characteristics of such composite material and better fabrication methods. Some of the fabrication methods, physical properties and microstructure characteristic have been reported so far and process needs further investigation for better understanding the characteristics of such matrix. In this paper, emphasis is given on are fabrication methods, physical behavior and probable application of nano composites reported so far and efforts has been made to indicate the future scope of study on nano ceramic composite.

Keywords: ***Ceramic composite, microstructure, Strengthening potential***

1. INTRODUCTION

Nanocomposite are materials in which at least one of the dimensions is in nano meter range. Reduction of reinforcement to nano range makes interaction of particles with dislocation more significant and leads to improvement in various properties which have proven to be useful for a wide range of critical applications. Extensive research is going on worldwide to improve the desirable properties of ceramics by adding reinforcements and limiting their inherent weaknesses. The Ceramic composites based on SiC, Si_3N_4, TiN, TiB_2, Alumina, Zirconia, TiC and many other have been developed and mechanical properties such as tensile, compressive, fatigue resistance, fracture toughness, R-Curve behavior, creep have been studied by various researcher for different CMCs. At elevated temperature better physical properties of CMCs are obtained as compared to monolithic composite due to nanoscale reinforcement (increasing the surface to volume ratio) which makes it suitable for ceramic cutting tools, wear resistive components, radiation resistive ceramic component, aerospace, & automobile components and other applications. Fracture toughness decreases when components are subjected to high temperature limits in some application, however, it is also reported that toughness improves with reinforcement in CMCs.

Main challenges are involved in synthesis of these materials which require advanced processing techniques. These challenges can be either due to the characteristic of reinforcing phase or limited processing techniques.

This paper is aimed at reviewing the fabrication methods, mechanical properties, strengthening mechanism and application in field of ceramic matrix composite reported by various investigators.

2. PREPARATION METHODS AND PROPERTIES

Self-propagating high-temperature synthesis (SHS) method is basically used for producing inorganic compounds by exothermic reactions, usually involving salts. Synthesis of nano-sized precursor powders is performed by special techniques like high energy ball milling process, sol-gel processing, gas condensation process, inert SHS reaction and infiltration technique. Consolidation difficulties of nano-sized powders, caused by their higher propensity to form strong agglomerates because of very high ratio of surface area to volume may be reduced by using various specialized techniques while their fabrication.

Infiltration methods are used to fabricate ceramic matrix composites reinforced with long fibers. This type of Ceramic matrix is formed with a fluid (liquid or gases) which is infiltrated into the fiber structure. The surfaces of the reinforcing fibers are coated with a deboning interphase prior to infiltration which weakens the bonds interface between matrix materials and the fiber. Weak bonding allows these long fibers to slide in the matrix and this results in the prevention of brittle fracture [1].

In Sol gel processing, sol is formed by dispersing the matrix and reinforcing particles in the liquid. The deposition of this sol solution results in coating on substrates by spraying, dipping or spinning process. Gel is formed from evaporation of solvents and particles or ions join together to form network. Thermal treatment is done to enhance mechanical properties. It is low temperature process and generates highly pure and well controlled ceramics [2].

Nano SiC/TiN composite was prepared by sol gel method using, TiN nanopowder as toughening phase, b-SiC nano powder as matrix phase and YAG (synthetic yttrium aluminum garnet) as sintering additive. Combination of aqueous slurry with spray-drying was used to prepare Nano SiC based granules. It was then uniaxially pressed at 160 MPa for 10s and pressed isostatically with a pressure of 250 MPa for 300 seconds [3]. TiC x/2009Al particles were ball milled at a speed of 100 rpm for 50 hours. Combustion synthesis was conducted in self-made vacuum vessel with a vacuum degree about 0.5 tar. Composites were extruded at 773 K under an extrusion ratio of 16:1 [4].

Al_2O_3/ AlN particle were ball milled in ethanol for 48 hours and then dried [5]. In Al_2O_3/TiC nano composite, the nano scale TiC powders were prepared into suspension using dispersant as polyethylene glycol and alcohol as the dispersing medium. After that, micro-scale TiC, Al_2O_3, and cobalt was added into the suspensions. After ball milling for 48 hours, dry type evaporator with vacuum was used for drying [6]. Alumina/zirconia/nano-TiO_2 ceramic composites were ball milled for 2 hours for mixing. The mixed powder was semi-dried and pressed at 100 MPa [7].

In ZrC/SiC composite, ZrC powder was first heated in air for about 10 hours at 250°C and mixed with SiC powder and pyrolysed under argon at 600°C for 5 hours and further ball milled [8]. In α- Al_2O_3/Si_3N_4 nano composite, α- Al_2O_3 and Si_3N_4 were mixed and ball-milled with ethanol for 72 hours and dried in a vacuum dry evaporator at 110°C [9].

Earlier most of the nano ceramic matrix composite were developed using hot isostatic pressing (HIP), hot pressing and sinter forging. Sintering kinetics is increased by the application of working pressure above atmospheric pressure. Limitation of conventional sintering techniques is the formation of strong agglomerates of nano sized powder due to extremely high ratio of surface area to volume which results in difficulty in consolidation leading to poor mixing, inhomogeneous packing, residual porosity and poor density.

With the advancement in fabrication technique today, most of the nano ceramic composite are fabricated by Spark Plasma Sintering (SPS). Heating rapidly to sintering temperature (Temperature lower than conventional sintering) and less holding time results in good control of the fine grain size, retention of nano scaled microstructure and high relative densities.

SPS of ZrC/SiC for developing ceramic composite was done at 1950°C temperature for 15 minutes under vacuum with a pressure of 50 MPa. The sample was then cooled to 1200°C with 25°C/min in order to reduce any quenching stresses. It was observed during sintering of the composite that overall strain associated to the applied load was preferably accommodated by plastic deformation of ZrC to a much lesser degree by the formation of stacking faults through phase transition operating within SiC. Sintering temperature in this process was high for retaining better properties [8].

ZrC/SiC Composite has been developed by solution based processing using divinylbenzene, polycarbosilane polyzirconoxane, to obtain ZS precursor prepared at 200°C and then heat-treated for 2 hours to a temperature of 1500°C with 5°C/min heating rate in argon atmosphere. Highly crystalline ZrC and SiC phases were observed in ceramic powders with 100–400 nm particle size. Distribution of Zr, Si, C was uniform at different sites in the powder. ZrC/SiC weight ratio was varied to control different element in the sample and precursors with good stability and processibility was used in polymer infiltration pyrolysis process. The prepared composite sample with less carbon content exhibited good oxidation resistance at high temperature [10].

In another method of fabricating ZrC/SiC Composite, tape-casting process and vacuum hot-pressing was used. In this method, ZrC and SiC powder was used as raw material for tape casting. First, sols of 5 wt% polyvinyl butyral resin (PVB) and 5 wt% polyethylene glycol were dissolved in ethanol as the adhesive and plasticizer, respectively. The mixture was placed in a water bath heated at 60 °C to obtain a homogenous material. Second, 20 wt% ZrC powder was introduced to the above mixture and dispersed in ethanol by ultrasonic agitation for 2 hours to form a homogenous mixture with a certain viscosity. Third, tape casting mould was used for placement of sol on it at room temperature for 8 hours and were cut into slices in the form of sheets**.** The same steps were carried out for SiC and then ZrC and SiC sheets were alternately stacked. The stacked sheets were heated at 550 °C for 60 min with heating rate of 10 °C/min to remove the binder. Finally, vacuum hot pressing furnace at 1700 °C was used for sintering the laminated sample for 90 min under an applied pressure of 20 MPa. Fracture behavior of laminated ZrC–SiC ceramics was quite different from brittle fracture and showed a non-catastrophic failure behavior. The crack deflection extended the crack propagation path and increased the energy consumption capacity of laminated ceramics and thus increased the fracture toughness [11].

Sintering of Al_2O_3/TiC was done between 1600ºC - 1700ºC in sintering furnace in vacuum for 10-30 minutes [6]. Wetting between metal interfaces and ceramic was improved by the addition of cobalt in the composite. Cobalt presence at grain boundary not only prevented TiC and Al_2O_3 from growing but also restricted the reaction between TiC and Al_2O_3 during the process, vapour phase formed during processing caused pores in Al_2O_3/TiC ceramics. At maximum sintering temperature cobalt liquefied and filled the pores in grains exhibiting better density. However, in Al_2O_3/TiC composite fabricated by Spark Plasma Sintering at different temperatures 1100 ºC, 1200 ºC, 1400 ºC, 1500 ºC for 3 minutes with 50 ºC /minute heating rate at a load of 60 MPa, complete densification was observed at lower sintering temperature than conventional sintering [12].

Another method of fabrication of Al_2O_3/TiC nano composite has been reported, using hot pressing. The samples

were prepared using alpha alumina, micro TiC, nano TiC and Cobalt. Firstly the TiC powder (nano scale) was prepared into suspension using dispersant as polyethylene glycol and alcohol as dispersing powder and subjected to ultrasonic dispersion for 20 minutes. P^H value of 9 was obtained by addition of $NH_3.H_2O$. It was then mixed and ball milled for 48hours and dried in dry type evaporator in vacuum. The dried powder was poured in graphite die and hot pressed with a pressure of 32 MPa at 1650°C in vacuum sintering furnace for 20 minutes. Composite showed better wear resistance and fatigue behavior to be used as tool material [13].

Alumina/zirconia/nano-TiO_2 nano composite sintered at temperature of 1600 ºC for 1 hour with 5 ºC /min heating rate from room temperature to 1000 ºC, and 2.5 ºC /min heating rate from 1000 ºC to 1600 ºC. Addition of TiO_2 formed Al_2TiO_5 ceramic which showed resistance to thermal shock [7]. However, high sintering temperature decomposed Al_2TiO_5 into Al_2O_3 and TiO_2 leading to increase in apparent porosity.
Two step pressureless sintering for fabrication of Nano-SiC/TiN nano composites reported in [3].Composites were first sintered at 1900ºC for 15 minutes in vacuum furnace and second step sintering at 1700 ºC, 1750 ºC, 1800 ºC and 1850ºC for 45 min at each temperature. YAG was used as sintering additive for densification. The densification in second step sintering was observed by slower grain boundary diffusion which restricted the grain growth. Y_2O_3 was used as sintering additive for fabrication of α- Al_2O_3/Si_3N_4 by hot pressing at 1450°C temperature in vacuum for 30 minutes under a pressure of 32 MPa. Due to the covalent bonding nature of Si_3N_4, it was sintered to a high density by addition of Y_2O_3 as sintering additive [9]. Al_2O_3/AIN composite was fabricated at 1600 ºC under an applied pressure of 30 MPa for 180 minutes [5].

Fabrication of nano ceramic composite by stir casting method is reported by few investigators. A resistance furnace equipped with inert gas injection instrument and a graphite stirring system was used to fabricate TiB2/ A356 Al nano composite at casting temperatures of 750 ºC, 800 ºC and 900 ºC. An increment of volume fraction of reinforcements and decrement of the particle's size lead to the increment of the porosity content. Decreased density and dislocation pile up phenomena was observed due to the presence of slip band in the matrix. Stir casting method of fabrication is not suitable for fabricating nano ceramic composite [14].

Zirconia toughened alumina nanocomposite was developed by SPS at 1100°C with heating rate of 500°C/min via combination of High Energy Ball Milling process followed by SPS of γ-alumina powders added with zirconia and yttria. Full densification was observed in nanocomposites which was SPSed at 1100°C. The hardness and toughness was increased which is almost three times than monolithic alumina due to addition of zirconia [15].

2.1 Mechanical Properties

Development of new material implies value-added to the physical properties. CMCs shows improvement in mechanical properties due to superior strength, hardness, abrasion resistance and chemical inertness in comparison to other materials. Reduction of grain size to nano meter range leads to hardness increment which improves mechanical properties as reduced wear behaviour, higher fracture toughness and higher resistance against abrasion. Physical properties of CMCs vary with fabrication techniques indicating that better the fabrication method better is the development of CMC material. The reduction of grain size to nano metric range of SiC in the composite ZrC/SiC fabricated by spark plasma sintering leads to an improvement of fracture toughness and flexural strength due to densification at high sintering temperature, the overall strain associated with the applied load was preferentially accommodated by the plastic deformation of ZrC [8]. The creep behavior remained unaffected upto 1600 ºC in Al_2O_3–TiC composites fabricated by the same method at lower sintering temperature showed homogenous distribution of titanium carbide in the alumina matrix, there was no new phase formations during sintering. The fully dense Al_2O_3/TiC composite showed higher Young's modulus and hardness values. Scratch test on the bulk sample showed that sample sintered at higher temperature had better scratch resistance due to strong bonding of the particle [12]. Addition of Cobalt in Al_2O_3/TiC fabricated by chemical deposition method revealed greater improvement in fracture strength of composite. Fracture toughness was also increased reducing the crack propagation [6]. Two step pressure less sintering of Nano-SiC/TiN nano composites densified the composite with improved properties as Vicker's hardness, bending strength and fracture toughness. Similarly addition of YAG in composite enhanced toughness by crack deflection [3]. Hot pressing of Al_2O_3/AIN composite at high temperature showed significant improvement in the properties like flexural strength, fracture toughness and relative density [5].

Hot pressed α- Al_2O_3/Si_3N_4 nano composite at lower temperature revealed optimum mechanical properties as flexural strength, hardness, fracture toughness and high relative density [9]. Increased in Si_3N_4 content beyond optimum value lead to crack formation on applied load which weakened the grain boundary strength and caused stress concentration. Flexural strength was decreased due to propagation of cracks easily. Sintering of alumina/zirconia/nano-TiO_2 ceramic composites exhibited higher density and less porosity. Hardness was significantly improved along with resistance to thermal shock [7].

The strengthening mechanism of Nano ceramic matrix composite is used to analyze the physical properties of the material. Orowan Strengthening Mechanism [16-18] has been used to analyze the effect of reinforcement and inter particulate spacing of secondary phase disperiods. Orowan strengthening is caused by resistance of particles to the passing of dislocations. Creep resistance raises considerably even for a small volume fraction due to dispersion of fine insoluble particle in the ceramic matrix. Ceramic reinforcement particle pin the crossing dislocation and assist dislocation in bowing around the particles. For simulation of better mechanical

properties of nano ceramic composite Orowan loop mechanism is used.
Yield strength σ_R of composite can be given by

$$\Delta\sigma_R = \frac{0.13\ b\ G}{d_p \sqrt[3]{\frac{1}{2V_p} - 1}} \ln\frac{dp}{2b} \quad (1)$$

Where b - Burgers vector
d_p -particle diameter
G - Shear modulus
V_p - volume fraction of reinforcement.

The Hall Petch Mechanism relates grain size with strength. Decreasing the grain size leads to increase in strength and ductility. Fracture resistance also generally improves with reductions in grain size. The yield strength of many metals and their alloys has been found to vary with grain size according to the Hall-Petch relationship:

$$\sigma_y = \sigma_i + \frac{k_y}{\sqrt{D}} \quad (2)$$

Where k_y - Hall-Petch coefficient (material constant)
D is the grain diameter
σ_y - is the yield strength of an imaginary polycrystalline metal having an infinite grain size.

Grain boundary play critical role in the yield stress of material. There can be several different deformation modes associated with different grain size, grain shape, temperature, stress state and grain boundary structures [16][19].
Dislocations can generate in the alloy matrix during processing due to coefficient of thermal expansion mismatch (CTE) between the matrix and reinforcement phase and induce residual stresses [16][20]

Mismatching of strain due to difference in CTE values of matrix and particles leads to the generation of thermal stresses at the interface which makes plastic deformation difficult leading to enhancement in flow stress and hardness. The effect of mismatch strain is given by

$$\Delta\sigma_{CTE} = \sqrt[3]{3}\,\beta G_m b \sqrt{\frac{12\Delta\alpha\Delta T}{(1-\nu_p) b d_p}} \quad (3)$$

where β - strengthening coefficient
α- difference between CTE of matrix element and reinforcement element
ΔT-difference between the processing and the testing temperatures
b-Burgers vector
G_m -shear modulus
d_p -particle diameter
ν_p -poissons ratio.

The Griffith's energy gives the basic explanation for the strengthening and toughening mechanisms of composite based on equilibrium between the fracture energy and energy release rate [21]. Rising R-curve behavior is observed in many ceramic based composites.
Crack resistance of this class of material is expressed by

$$K_R(\Delta\alpha) = K_i + \Delta K_R(\Delta\alpha) \quad (4)$$

Where K_R (Δ α)-fracture toughness of the material which shows R curve behavior
K_i- intrinsic fracture toughness
ΔK_R (Δα)- extrinsic increase of fracture toughness after a definite extension beyond the initial crack tip Δα.

Griffith–Irwin formula for materials with an R-curve is given by

$$\frac{K_R^2}{E'} = 2(\gamma_i + \gamma_R) \quad (5)$$

γ_i and γ_R are the intrinsic and extrinsic fracture energy per unit area of the cracked surface, respectively.
Frontal process zone (FPZ) ahead of crack tip is composed of nano cracks rather than dislocations. Fracture toughness can be increased by expanding the size of FPZ. The left side of Griffith–Irwin equation indicates release rate of critical energy beyond a definite crack extension in materials having R-curve behavior.

3. APPLICATIONS

Production of ceramic matrix composite using nano technology can be made more useful, cost effective and high ending in the service conditions. Nano ceramic matrix composite is used in variety of application based on its structure, properties, strengthening and toughening mechanism. Some of the applications of various ceramics are discussed in this section.

The Al_2O_3-based composite is used as tool materials for high speed machining compared to the traditional cemented carbide cutting tools and high-speed steel because of the good mechanical properties as high hardness, high corrosion and wear resistance [22].

Nickel based alloys are typically being used in high pressure turbines, mobile phones, medical equipment, transport, buildings and aerospace application due to its better corrosion resistance, toughness strength at variable temperatures. Materials such as titanium and nickel based alloys having properties of light weight and high strength to weight ratios are highly demanded in aerospace, automotive and power industries for their critical components [23].

Ceramics based on the carbides of the Group 4 transition metals are of great interest for applications at high temperatures in both aerospace and nuclear applications [8]. SiC nano composite are used in applications requiring high endurance such as car brakes, car clutches and ceramic plates in bulletproof vests [24].

Aluminum oxide referred to as alumina are used in structural applications. Alpha phase alumina is stiffest and the strongest among all oxide ceramics having high hardness, high refractoriness, excellent dielectric and thermal properties

which makes it a suitable choice for different applications such as in manufacturing of wear pads, grinding media, seal rings, high temperature electrical insulator and aerospace components [25].

Alumina based ceramic, (ZrO_2) toughened improves the toughness and resistance to fracture and are used as tool material [26]. Zirconia (ZrO_2) based ceramic material with adequate mechanical properties are used for manufacturing of medical devices [27].

Alumina Mullite ceramic is used as a traditional and advanced ceramic material because of having favorable thermal and mechanical properties for structural applications [28].

Silicon nitride (Si_3N_4) ceramic material having excellent thermo mechanical property is most suitable for structural applications, bearings, cutting tools and engine components. It performs better at high temperature retaining high strength and creep resistance and low thermal expansion coefficient giving good thermal shock resistance [9]. Titanium based ceramic are used in the manufacturing of wear-resistant tools, cutting tools and coating for abrasive steel bearings [29].

4. CONCLUSION

In this paper effort has been made to understand the CMCs process of development and its characteristics. The application of various CMCs literature report has been discussed and following conclusions have been made.

Spark Plasma sintering is found to be most advanced technique for fabrication of CMC which restricts the grain growth and densify the material at high temperature providing better strength.

Effect of various phases play an important role in internal stress distribution within the composite affecting the properties. Mechanical behavior of nano composite is presented which showed strength and hardness increased by nano particle reinforcement. However, flexural strength is decreased in some cases with the increase in reinforcement percentage.

Exclusive study and development of better CMCs fabrication techniques are needed for improving overall properties to be used for wide range of application.

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