

 ISSN NO. 2320-5407	Journal Homepage: - www.journalijar.com INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR) Article DOI: 10.21474/IJAR01/12794 DOI URL: http://dx.doi.org/10.21474/IJAR01/12794	 INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR) ISSN 2320-5407 Journal Homepage: http://www.journalijar.com Journal DOI: 10.21474/IJAR01
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RESEARCH ARTICLE

CARBON NANOTUBES REINFORCED ZIRCONIA COMPOSITES FOR ARTIFICIAL HIP JOINT'S BEARING SURFACES

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Manuscript Info

Manuscript History

Received: 25 February 2021

Final Accepted: 30 March 2021

Published: April 2021

Key words:-

Totalhiparthroplasty, Carbonnanotubes, Zirconia, Ceramic Composite, Femoral Head.

Abstract

This research focuses on the properties and capabilities of carbon nanotubes (CNTs) as reinforcing agents in ceramic composite bearing components. The addition of CNT to pure ceramics can form composites with much better performance than conventional ceramics for various applications. The study was started from the pure ceramics in their use as a component of bearing total hip arthroplasty and the damage that occurred after implantation. The study covers factors that cause degradation of conventional ceramics; introduction of properties and use of CNTs as new reinforcing agents for ceramic composite materials; widely used sintering method; and the heat and pressure parameters used during the sintering process to meet the standards for the manufacture of Total Hip Arthroplasty (THA) bearing component. The contact and buckling behaviors of the CNTs influence the composite's friction properties. The presence of CNT until 20 wt% of CNT exhibited improve wear resistance with lower friction with the increase of weight percent. CNT reinforced ceramic coating might be capable to withstand high load-bearing conditions. Plastic deformation can be one of the critical processes in wear in the ceramics wear mechanism. Other processes are cracking and chemical reaction. The microstructures and porosity take an important role in indicating the ceramics wear properties and wear mechanisms.

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Introduction:-

This review article presents the carbon nanotubes (CNTs) role in reinforcing ceramic composite materials development. Some literatures havereported that the addition of CNTs to pure ceramics can form composites with much better performance than conventionalceramics for use in various applications, includingtotal joint implants (hip and knee). The research has started from the literature study of pure ceramics total-joint implant bearing surface; factors influencing the THAs' ceramic component durability; types of degradation occur on pure ceramics bearing components; introduction of CNTs agent properties; preparation technique for CNT-reinforced ceramic composite materials, the selection of the sintering method, the study of the heat and pressure parameters used during the sintering process to meet the standards the manufacture of THA femoral heads, and cup liners application. This

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research activity received financial support from the Ministry of Research and Technology / National Agency for Research and Innovation and BPPT of the Republic of Indonesia.

Carbon nanotubes (CNTs) reinforced ZrO₂ composites have been intriguing the interest of researchers and technologists to work on due to their ability to self-heal from crack and the possibility to tailor the desired nanostructured properties. In the research and development of ceramic composites with nano-fillers, just a few new technologies and new processing methods have been founded and implemented. A clear understanding of the wear response and tribological features of the CNT/ceramic composites, such as wear mechanisms, friction followed by wear rates evaluation in bio-tribology of bone implants, such as total hip joint arthroplasty, has not been well-documented in the literature [1]. Regarding the CNT/ceramics composites, the relationship between reinforcing and matrix weight fraction and preparation technique is essential to take into consideration. They could affect the composite's microstructure, the mechanical and tribological properties of the materials [2].

Total hip arthroplasty (THA) plays a significant role for people who suffer from the hip-joint disorder due to, for example, degenerative hip bone, accident, osteoarthritis, hip bone joint cartilage worn, and so forth [3]. The number of total joint arthroplasty users has grown significantly worldwide and expected to rise over time. Four main types of bearings available in THA application are ceramic-on-polyethylene (CoP), metal-on-metal (MoM), metal-on-polyethylene (MoP), and ceramic-on-ceramic (CoC). Recently, the introduction of hybrid combinations used for ceramic heads and metallic inserts (CoM) has been reported in published works [4, 5, 6, 7, 8]. Some factors that affect the choice between these types of bearings are the implant cost, patient age, patient level activity, surgery complications level, etc. The presented review focuses on reported research delivering the preparation and testing of both pure and improved (composite) zirconia ceramic bearing surface of femoral heads [9], including things related to tribological aspects in THA's system.

To obtain a well-presented literature study presents a compilation of published works with the following arrangement: (1) select and define the discussed topics. (2) Search research topics on the internet; (3) Selecting literature for the related topics of interest; (4) Studying the preparation methods reported in the literature; (5) Showing examples of SEM/TEM micro or nano-graph images taken from the literature; (6) Reviewing analysis results made by authors, the correlation between observed micro/nanostructure images and materials mechanical properties; (7) Providing a brief conclusion or summary regarding micro/nanomaterial's structures and composite materials mechanical properties, for a particular method of materials preparation used and applications.

Zirconia ceramics have been used for a very broad application range, such as cutting tools, fuel cells, dental implants, bearing components, sensors and so on. In the medical sector, the use of Zirconia ceramics has been common due to its exceptional mechanical properties and biocompatibility [2]; Zirconia has been used as material for making femoral head as a component of the THA because of its properties, which had strength against impact loads and excellent wear resistance. Zirconia femoral heads offer improved fracture strength. Hence, Zirconia femoral heads are the most favorite material for THA. However, it was found from the investigation that the ceramic femoral head failure because of aging. The incident of the ceramic femoral head failure resulted in a catastrophic impact on the patient's choice for zirconia in the medical sector [10]. Some researchers believed that transformation of monoclinic phase after implantation is still a potential mechanism for the failure of ceramic head [2].

Methodology:-

This paper is a literature study based on journals, conference papers, and other relevant publications. To find appropriate journals, conference papers, and other published works, they were searched from the internet using the keywords, namely total hip arthroplasty, carbon nanotubes, zirconia, ceramic composite, and femoral head. The assessment of collected published reports is aimed to choose the relevance with the scope of this study. And the last they were summarized and synthesized to write this paper.

Results and Analysis:-

Zirconia Ceramics

In its natural form, Zirconia or zirconium dioxide (ZrO₂), is obtained mostly from the mineral baddeleyite [11]. However, chemically it can also come from zircon derivatives. Product characteristics that are made from zirconia are usually having excellent mechanical properties such as high temperature stability, strong resistance to corrosion and thermal, consistently high quality and chemical inertness. These characteristics are ideal for usage in wide varieties of

applications, including medical products, such as implants, due to their excellent biocompatibility for prosthesis devices. Some technical characteristics of hip joint femoral head made of zirconia ceramic are scratching resistance, good hardness and strength, stability and wear resistance, and its human body biocompatibility [2].

A researcher assumed that today implanted ceramics, especially zirconia femoral heads in total hip arthroplasty, mostly work very well and that only a little percentage of patients have experienced problems. In the world, zirconia femoral heads that have been used were around 600,000, mostly in Europe and USA. Since 2000, more than 400 or about 0.67% of failure following implanted zirconia femoral heads, have been documented. Publications pertaining with the initial diagnosis of the fractures of ceramic head have not been found yet. Later on, the presence of zirconia femoral head fragments larger than $5\text{ }\mu\text{m}$ following inspection of synovial fluid, intensely related to the presence of liner fracture was reported by Toni et al. [10]. In their research Francesco Traina et al. [12] confirmed the observation for bigger populations; the microanalysis of synovial fluid appears as a supportive instrument for initial diagnosis of zirconia liner fracture. Some researchers urged that the key issue pertaining to zirconia is intrinsic brittleness. The zirconia toughness can hinder deformation of plastic under loads. On a load of cyclic is given above the zirconia femoral head components, microscopic defectiveness of the material e.g. inhomogeneity or pores can turn into stress risers which causes the crack propagation so there is a chance of failure potential of the femoral head components [1, 2].

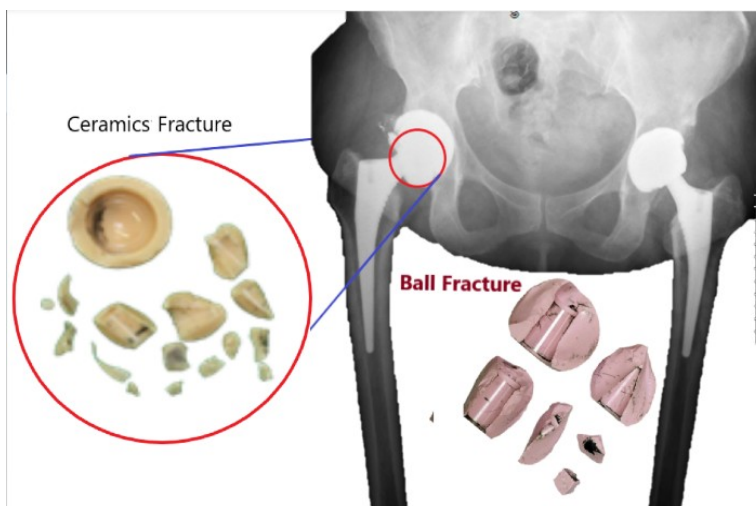


Figure 1:- Preoperative radiograph displaying failure and the retrieved Zirconia bearing [13]

Zirconia Structure

Zirconia shows three different types of crystallographic structures related to its temperature [14]. From room temperature until $1175\text{ }^{\circ}\text{C}$, it transforms into monolithic structure (m); at intermediate range of temperatures $1175\text{--}2370\text{ }^{\circ}\text{C}$, it appears in a tetragonal structure (t) and at the temperature is $2370\text{ }^{\circ}\text{C}$ or higher, it becomes a cubic structure. The transformation matches to changed characteristics which have exceptional wear resistance, excellent durability, and high component and flexural strength [15]. Zirconia in the tetragonal phase has several useful applications, including structural ceramics in physically demanding applications. If the temperature escalates over $2,370\text{ }^{\circ}\text{C}$, zirconia transforms into its cubic state.

In the cubic form, zirconia is famous as a gemstone because it has high refractive index and optical properties clear single crystal, in addition to its proficiency to keep its color and brilliance. It also is commonly known as a synthetic diamond. In the natural form, Zirconia can be found in the mineral baddeleyite or obtained chemically from zircon. Commercially it is considered to be the most valuable oxide obtained from zircon [16].

The techniques applied to extract zirconia from zircon are as follows [17]:

1. The zircon decomposition is involving thermal, chemical or mechanical means;
2. The decomposition of zircon is then treated using differentiation of solubility;
3. These entirely involve the separation of compounds of zirconium from residual impurities.
4. The production of zirconium oxide (fused zirconia) is in the way of the zircon sand reduction and fusion of (zirconium silicate). The mixing of zircon and coke by increasing its temperature to its point of fusion (above $2,800\text{ }^{\circ}\text{C}$) in an electric arc furnace will separate fumed silica and zirconium oxide [18].

Transformation Toughening

A research group has developed the transformation toughening phenomenon of zirconia ceramics theoretical models. The transformed process zone development is correlated with a progressing crack in which a change of the metastable phase (t) happens at the crack tip [19].

The tensile stresses could generate a transformation of phase from t to m surround the crack tip and causing a volume increase. The increasing volume triggers compressive stresses surround the crack and ceases for crack propagation further [20, 21, 22].

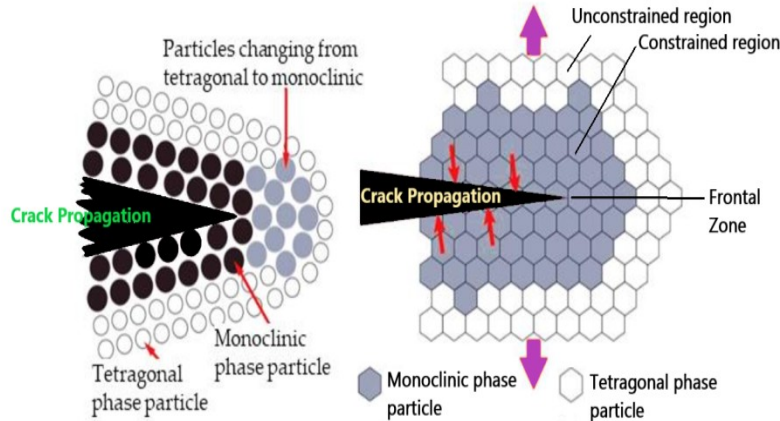


Figure 2:- A toughening of transformation example in front of crack propagation [23].

A phase transformation toughening model with linear elastic fracture mechanics has been developed by McMeeking and Evans in 1980 [24]. Modeling satisfies the truth that the stress-induced transformation toughness creates protection from the factor of applied stress intensity, meaning that the factor of actual stress intensity at the crack tip is smaller than that exerted by outside forces as shown in the equation below (equation 3.1):

$$K_{l_{tip}} = K_l - K_{l_{sh}} \quad (3.1)$$

where $K_{l_{tip}}$ is the factor of stress intensity at the crack tip, K_l is the factor of applied stress intensity and $K_{l_{sh}}$ is the shielding factor.

Preceding publications expose that the more increasing the factor of applied stress intensity, the bigger the zone of transformation and the bigger effect of shielding as presented in the below equations (3.2) and (3.3):

$$K_{l_{sh}} = C_{sh} K_l \quad (3.2)$$

$$C_{sh} = \frac{0.214 \cdot E \cdot V_f \cdot e^T \cdot (1+\nu)}{(1-\nu) \sigma_m^c} \left(\frac{\sqrt{3}}{12\pi} \right) \quad (3.3)$$

where E is the modulus of elasticity, V_f is the transformable particle volume fraction, e^T is the dilatational stretch related to the transformation, ν is the Poisson ratio and σ_m^c is the critical stress leading to the transformation of phase.

The zirconia toughness is depending straightly on the critical local stress related to transformation of phase σ_m^c . Also, the higher the σ_m^c , the lower the C_{sh} , and the contribution of C_{sh} will be small for high critical local stress. The transformation of local stress hinges on the how much of the undercooling temperature below the T_o (t/m) temperature. The more the undercooling below the T_o (t/m) induce higher critical stress for the transformation of stress-assisted phase, then the smaller the toughening of transformation [19, 25].

Zirconia Degradation

Low thermal degradation (LTD), also called as degradation or aging of hydrothermal, was first informed in 1981 by Kobayashi et al. [26]. It is stated that t-m transformation is slow on the surface in a humid condition at a temperature of 250 °C, samples of zirconia could experience microcracking and strength loss. Many other researches tried to

recognize the LTD phenomena since its discovery, but it is still under discussion [18, 19, 27]. LTD was considered vitally simply at the above the human body or room temperature (37 °C), following several hundredsof zirconia femoral heads failed when implanted in patients in a short time period after implanted. This disaster restricted the utilization of zirconia for medical applications so that deeper examinations were required to know LTD of zirconia [9].

In the course of LTD, the conversion happens from the phase of tetragonal to monoclinic. The achievement of the reversible conversion (m-t) will happen before an entire strength recovery of aged specimens at high temperatures annealing [28]. Yoshimura had conducted experimental observations on Zirconia LTD, the results were as follows [29]:

1. LTD acceleration occurs more rapidly at the range of temperature between 200°C to 300 °C, and this is time-dependent
2. In the form of micro-and macro-cracks, LTD appears as the t-m transformation result.
3. The transformation process takes place from the surface to the sub-surface of samples.
4. Water or water vapor boosts the transformation t - m to occurs.
5. The transformation resistance could arise by decreasing the size of grains and increasing the stabilizer.

The LTD mechanism is water and Zr-O-Zr links reaction at the flaw tip during the Y- and Ce-ZrO₂ alteration [30]. The observations reported that water reacts primarily with Zr-O-Zr bonds on the surface and not the stabilizing oxide [9].

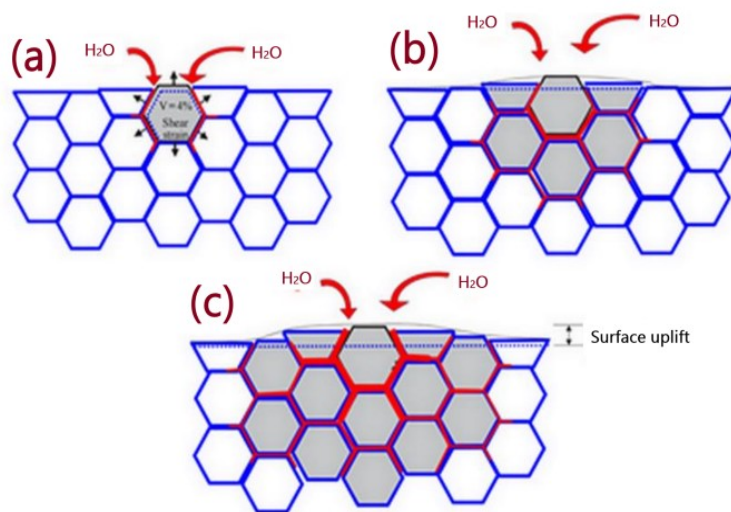


Figure 3:- Sketch describing the neighbor to neighbor transformation (a), the sub-surface nucleation to the top (b) and (c) sub-surface grain growth of the transformed zone [16].

Some research groups suggested that many factors that influence the LTD are mainly due to physical and mechanical characteristics of materials, e.g. grain size, density, phase distribution homogeneity, and surface residual stresses. Open porosity on porous materials may cause molecules of water to enter the bulk causing microcracking and failure of the materials [31, 32]. To improve the mechanical properties of the Zirconia, some researchers introduced Yttria Stabilized Zirconia (YSZ) [26, 32, 33].

Yttria-Stabilized Zirconia Structure

Yttrium oxide (Y₂O₃) and cerium dioxide (CeO₂) are considered as the best favorable stabilizers. At room temperature, the tetragonal phase is in the form of tetragonal zirconia polycrystals, in which the microstructure comprised of equiaxed fine-grains is preserved [34]. The oxygen overfilling around the small zirconium Zr⁴⁺ cations is accountable to influence the less stability of tetragonal zirconia. Hence, in order to create tetragonal zirconia stability, the usage of bigger trivalent cations (Y³⁺) size will be more efficient compared to the smaller size [20, 19].

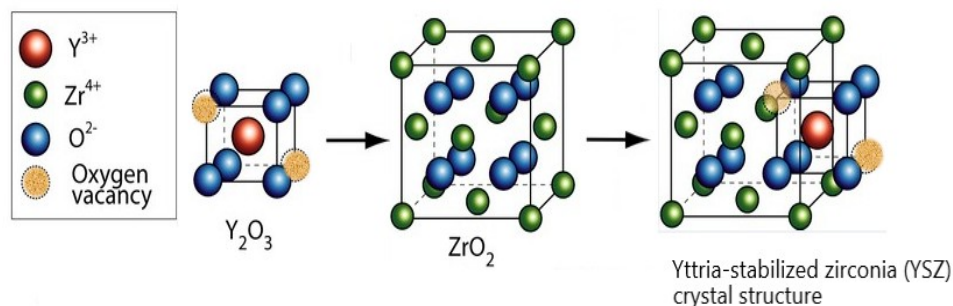


Figure 4:- Illustration of the three ZrO_2 polymorphs, according to space groups: (a) cubic (b) tetragonal and (c) monoclinic [35].

The presence of volume expansion is usually an indication of the tetragonal to monoclinic phase transformation. Some authors believe that the 0.05 volume growth followed by the microcracking state is related with the transformation of t-m phase, which commonly commences at around 950 °C on chilling in pure zirconia and is reversible heating about 1150 °C [18]. Some oxide dopants assimilation, for instance: magnesium oxide, calcium oxide, cerium oxide, and yttrium oxide, can repress the transformation of phase and become stable zirconia in the form of tetragonal or cubic. The stabilized zirconia types found reliant on the final microstructure shape reached, with a fully cubic structure. Meanwhile, Partially Stabilized Zirconia/ PSZ consists of the main phase, i.e. cubic zirconia and the trivial phase, i.e. monolithic and tetragonal zirconia precipitation. Tetragonal Zirconia Polycrystals / TZP contains a state, taken in a metastable state, up to room atmosphere [20].

Yoshimura et al. [36] reported the degradation in Y-PZP due to the OH molecules penetration could occur with the following steps: (1) started from chemical-adsorption of H_2O at the surface; (2) Zr-OH and/or Y-OH bonds formation at the surface; (3) OH migration; (4) OH^- ions migration at the surface and the lattice; (5) nucleation of monoclinic phase in the tetragonal grains.

According to Chevalier et al. [18] LTD experiences a mechanism of nucleation and growth. The transformation of nucleation in one grain causes an increase in volume that forces the grains to come into contact directly and subsequently spreads to other grains, triggering stretching between grains in the form of microcracks. And these cracks or gaps provide a means of for water to infiltrate the specimen. The extension of the transformation happens, especially in the neighboring grains. Nucleation usually occurs in the most unstable granules that experience the maximum internal/applied tensile stresses. The grain stability can be achieved by the presence of small or large size of Y_2O_3 experiencing higher internal stress.

Yttria-stabilized zirconia femoral heads in vivo degradation have been associated with the rise of surface roughness and even fracture. The degradation of micro-hardness of yttria-stabilized zirconia heads could also be related to age. However, the connection was not well identified.

Following the yttria-stabilized zirconia wear behavior, there are many proven experiments, indicating that the performance of tribology of 3Y-TZP on 3Y-TZP mating surfaces shows a high wear rate compared to the other ceramic pairs. Some research groups have investigated that the weakening of zirconia mechanical properties is associated with surface fracture induced by micro-cracking occurring during the transformation of phase from tetragonal to monoclinic (t-m) [37] and due to the low thermal conductivity of zirconia, causing a considerable rise in the contact zone temperature [38].

Growing interest in the development of advanced ceramic composites has increased in the last decade. The development purpose is to improve its mechanical and physical properties through integrating whiskers-like particles into the matrix of ceramic. In recent years, the carbon nanotube discovery has made significant interest in its usage as reinforcements or functionalizing elements in diverse materials, one of them is ceramics because of their high tensile strength (~100 GPa), high Young's modulus (~1500 GPa), high aspect ratio, high electrical conductivity and fine thermal conductivity [16, 34]. Single-wall nanotubes (SWNT) and multi-wall nanotubes (MWNT) using dissimilar processing methods have been done for the CNTs/ceramic composites preparation [16, 18] encompassing sintering and post-hot isostatic press (HIP) [19, 22, 39]. Recently, to minimize the damage risk of the carbon nanotubes, most

research works utilize spark plasma sintering (SPS) as their preparation method. This method runs using a low temperature of sintering, hot press, and short time of sintering [39].

Some authors informed the better properties mechanically and functionally for the zirconia ceramic/CNT composites contrasted with the monolithic material. Nanocomposites of CNT/ceramic show thermos elastic properties. Zirconia (3Y-TZP) is one of the materials used for many structural applications because of its mechanically high properties. Zirconia and zirconia-based composites are multipurpose materials used in various applications, for example solid oxide fuel cells, ceramic membranes and oxygen sensors because of their stability in elevated temperature, electric field with high interference, high energy bandgap, etc. The purpose of the presented review is to study the processing route of techniques that could be performed in future research on the development of CNT reinforced zirconia, for total joint arthroplasty, by taking advantage of results published research works in kinds of literature [25, 40].

The addition of CNT/3Y-TZP nanocomposite utilizing spark plasma sintering process affects the significantly grain size reduction, resulting in a decrease of t-m transformation; meanwhile, if the CNT's percolation arises with relatively little volume-fraction [40], the higher thermal conductivity can also affect the behavior of wear. Hence, Latifa Melk et al. [41] then investigated CNTs' effects on the composite of monolithic 3Y-TZP and 3YTZP/CNTs in terms of tribological performance and wear properties of [41].

Carbon Nanotubes (CNTs)

Many researchers believe that CNTs will be the key to the generation of advanced technology implemented by the next industrial era. Their characteristics are significant to the high demand for high-quality products. CNT has a cylindrical form constructed by a rolled plane of carbon nanotubes built with nanometric scale diameter and several centimeters length; CNT leads toward high aspect ratio with diameter length higher than 107.

Observation of multi-walled carbon nanotubes (MWNTs) for the first time in 1991 was conducted by Iijima[42]. Later in 1993, he found single-wall carbon nanotubes (SWCNTs)[43]. Figure 5 describes types of carbon nanotubes. A carbon nanotube (CNT) is a rolled carbon (graphene) plane in a cylindrical shape. The carbon nanotubes available with a high Young's modulus, the high tensile strength of ≈ 1500 GPa, and ≈ 100 GPa, respectively, considerably higher than carbon fibers and steel, higher than diamond in high thermal conductivity, and similar to silver and platinum in electrical conductivity. The CNT density is much lower than aluminum[39, 44].

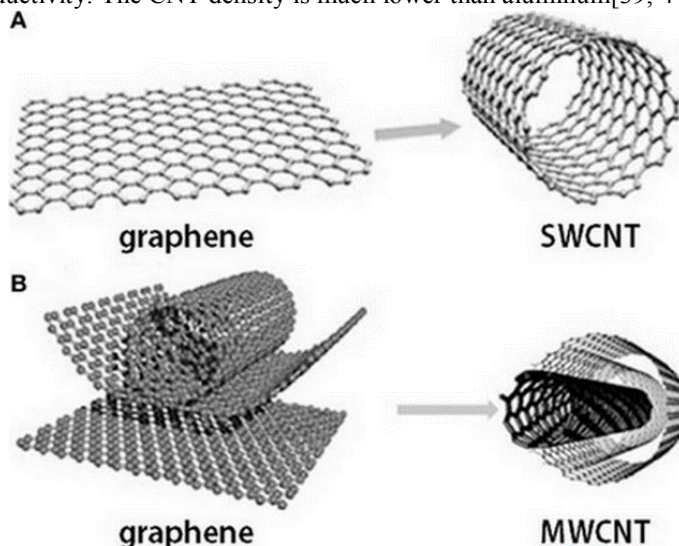


Figure 5:- CNTs structure according to their graphene cylindrical layer number:

(a) capped single-wall carbon nanotube (SWNT); and (b) open multi-wall carbon nanotube (MWCNT) [44].

The number of graphene cylinders (x) determines the name of CNTs. They denoted as single-wall CNTs ($x=1$), Double-wall NTs ($x=2$), and multi-wall CNTs (MWCNTs) with x is greater than 2. For MWCNTs, the distance between the two CNTs layer is around 0.34 to 0.36 nm, and the bonding span of carbon nanotube is 0.14 nm [44].

SWCNTs could cylindrically form with one sheet of carbon enveloped, and the cylindrical carbon sheet diameter is less than 2 nm.

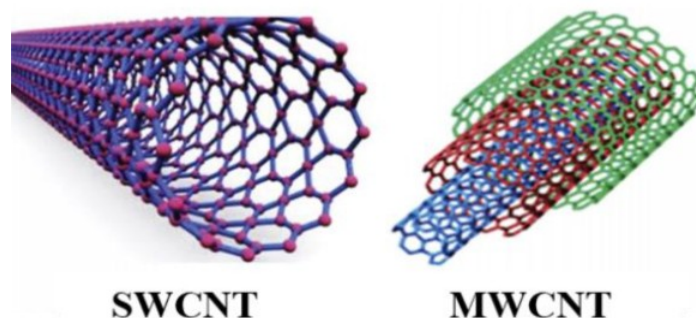


Figure 6:- Schematic diagrams showing different CNT types: Single-wall and multi-wall Carbon nanotubes(SWCNT and MWCNT) [40, 45].

Zirconia – CNTs Composites Processing

CNT Dispersion in Ceramic Matrix

Spreading of CNTs is the first step to obtain independently separated nanotubes that can lean either as fiber in one dimension, flat sheet in two dimensional, or bulk in three-dimensions. The spreading can be physically, mechanically or chemically. The mechanical spreading part involves a tube separator using ultrasonication, mixing at the molecular level, synthesis of sol-gel, and ball milling techniques. The chemical dispersion method is the technique to prepare a solvent or surfactant to reduce the surface energy of the nanotubes [46].

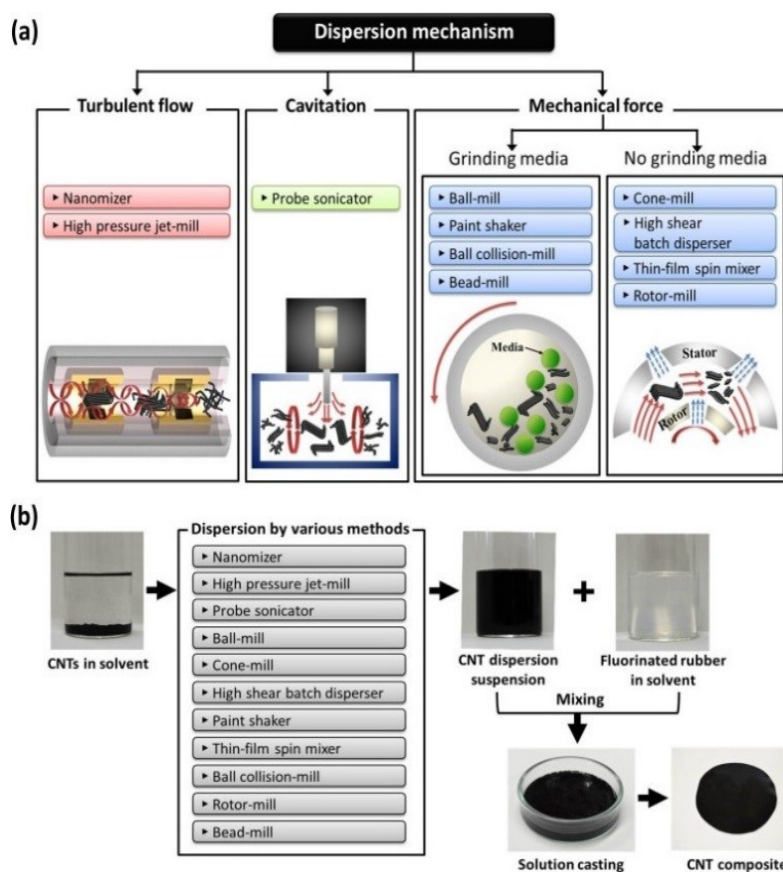


Figure 7:- Dispersion mechanism [47].

The nanotubes may increase their bonding features to the medium and decrease particles' clusters. Meanwhile, high chemical concentration or mechanical dispersion will affect the changes in carbon nanotubes characteristics [47]; the ultrasonication technique is to simplify the carbon nanotube dispersion mechanism by reducing the length of tubes. There will be, however, a threat of damaging the wall of tube. Accordingly, to anticipate such a case, an advanced ultrasonication method utilizing diamond crystal has been developed. With the developed method, some believe that although the SWCNTs rolls are ruined but not happening to the nanotubes. A research group reported that the ultrasonication appears to rip the outer most sheets of the MWCNTs, and it will get length reduction and thinner over time [40]. Ultrasonication can then be an impurities removal. Ultrasonication-assisted filtration studies exhibited that carbon nanotube purification were from $\approx 70\%$ to $\approx 90\%$ [48, 49].

Aside from the dispersion techniques, CNTs can be directly developed on ceramic nanoparticles by CVD. The limitation of CNT dispersion is in the matrix of ceramic and the fraction of CNT weight. The fraction of CNT weight has to be below the highest required weight fraction to avoid the agglomeration formation of CNT, which then causes a decline in the strength and toughness of the CNT / Ceramic composites [49].

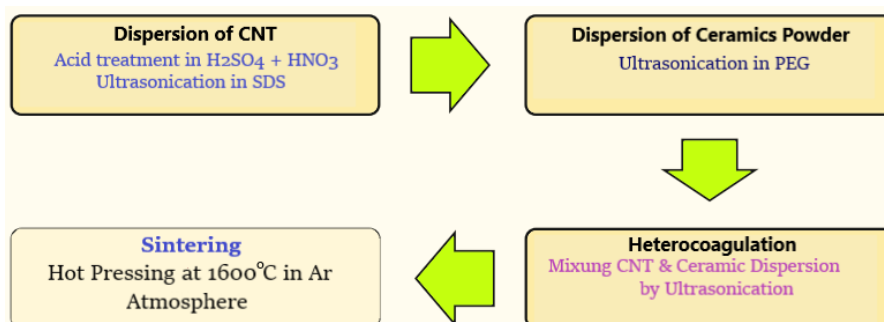


Figure 8:- Flow chart of CNT-reinforced ceramic composite synthesis by ultrasonic dispersion and heterocoagulation [49]

Sintering

Sintering is a thermally compacted powder process to produce dense polycrystalline materials at a temperature just under the melting point condition. The sintering is thermodynamically an irreversible route involving free energy reduction with surface energy decrease. Methods of sintering are mainly using the following techniques: (a) Hot Isostatic Pressing (HIP) [44, 45]; and (b) Spark Plasma Sintering (SPS) [50, 51];

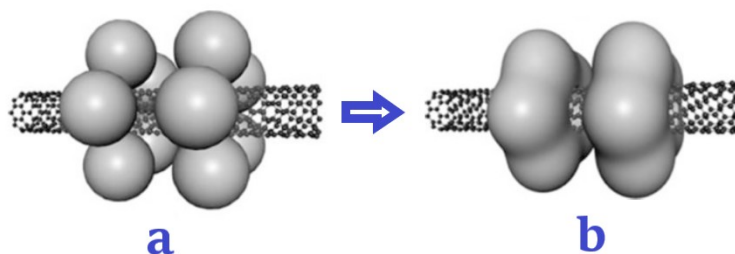


Figure 9:- Implantation of ceramic particles on CNTs. (a) before dan (b) after sintering [52]

Hot Isostatic Pressing (HIP)

The Hot Isostatic Pressing (HIP) technique is bonding nuclear fuel element assemblies diffusion [53]. With HIP, materials' bonding could occur in a high-temperature environment and isostatic pressure. This is what is named Gas Pressure Bonding [54]. HIP could produce high-performance and high-temperature ceramics such as silicon nitride and silicon carbide. Furthermore, the HIP is operated to produce alumina for cutting tools with economical but superb cutting properties. HIP could offer many advantages. Some of them are high densification, porosity elimination, mechanical properties improvements, such as fatigue, creep, ductile and strength, and recovery of defective parts. Besides that, the HIP can reach the preferred form with a high control and accuracy level [55, 56]. HIP apparatus equipped with an electric oven installed in a pressurized container. HIP apparatus contains a pressure vessel electric oven. The high-pressure spreads argon gas into the pressure container. The use of argon gas is due to

the argon gas has very low viscosity and very high density. Accordingly, the natural gas convection could transmit the generated heat. The HIP chamber consists of a furnace made from heat-resistant materials or refractory metals [55].

Spark Plasma Sintering (SPS)

The Spark Plasma Sintering (SPS) machines are used by Sumimoto Coal Mining Company in its industrial production. The production commencement was based on DC pulse to heat the loaded sample. Similar equipment based on DC. Today, more than 1750 units of SPS machines have been installed worldwide, with two-thirds of the related industry. More than three thousand publications have been using the SPS technique [50].

The SPS configuration contains a two-directional pressure structure, installed with an electrical circuit situated in a controlled compartment. A direct current pulse originator, water-conditioned reactor chamber, a pressure-, heat-, and site-variable arrangements installed in the device. During the sintering process, the powder poured into the die is mechanically pressed [50], as seen in Figure 10.

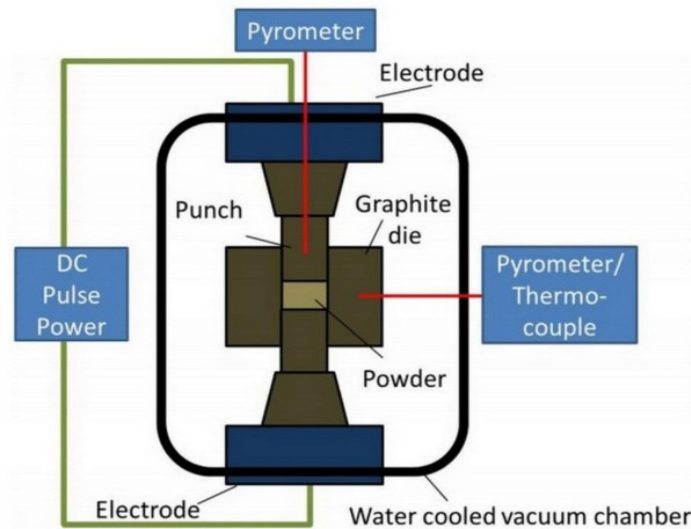


Figure 10:- The SPS process schema for the different parameters involved during sintering [50]

Figure 11 shows the SPS process schema. The sintering machine consists of punch electrodes, a water-cooled vacuum chamber, a uniaxial press, controlled atmosphere, generator and direct current pulse position, temperature and pressure measuring parts.

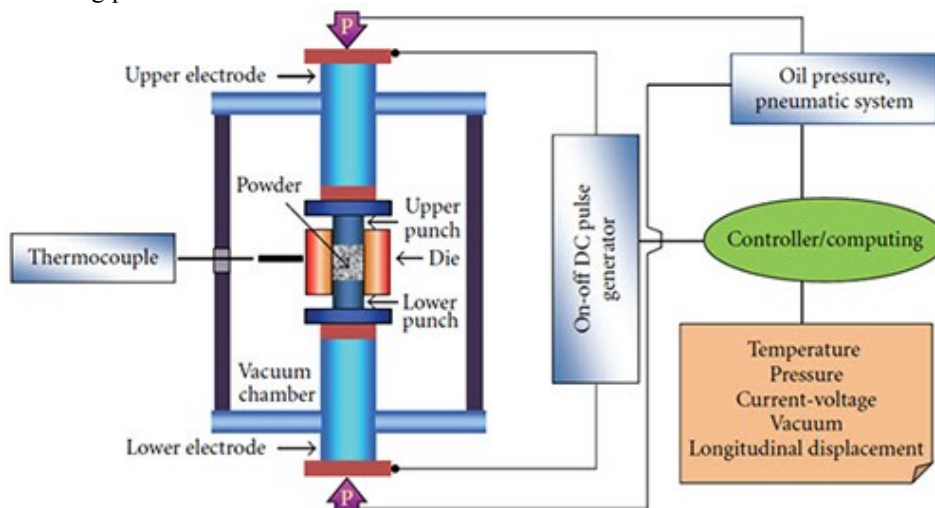


Figure 11:- Schematic of SPS process [57]

The temperature of sintering might be controlled by setting the ramp rate, elapsing time, pulse duration, and pulse current and voltage. Generation of spark plasma, spark impact pressure, Joule heating, and an electrical field diffusion effect could be initiated by the DC pulse discharge. In SPS, sintering is assisted by the on-off DC pulse voltage compared to conventional hot has shown in Figure 8. The application of pressure helps the plastic flow of the material. Figure 11 and Figure 12 schematically illustrate the flow of DC pulse through the particles [58, 59].

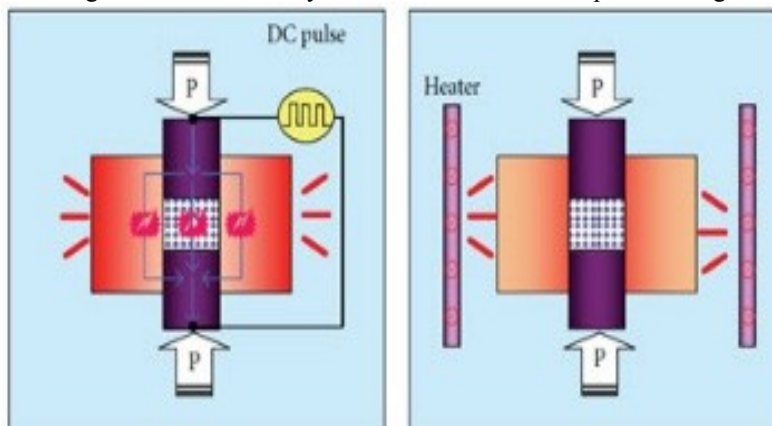


Figure 12. Comparison between SPS and conventional sintering [58].

The SPS consists of four main working steps. The first step is to eliminate gases to make a vacuum. In the second step, the pressure application is followed by resistance heating in the third step, and in the last one is a cooling down implementation. When an outflowing spark appears in the gap or at the point of contact between material particles, there will be a very high heat generation, ranging from tens to thousands of degrees of centigrade in a short moment.

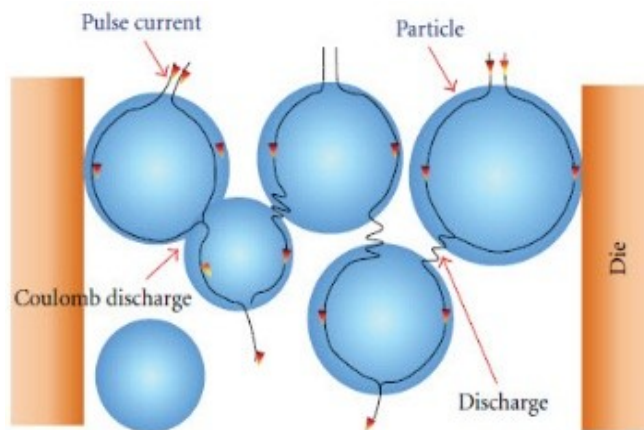


Figure 13:- The direct pulse current flowing through the particles [40]

In the SPS process, evaporation and melting occur on the powder particle surface, and also the necks formation around the contact areas between the particles, as shown in Figure 13. Besides the generation of high localized temperatures via resistance pulse heating, the pressure application and current can increase the heating rate and decrease sintering time and temperature leading to consolidation of nanopowders without excessive grain growth. Meanwhile, the SPS process is binderless and does not have a pre-compaction stage. The powder is poured directly into the graphite mold to be pressed under an electric current field to obtain a highly dense material with superior mechanical properties [41, 58].

CNT Effect on the Ceramics Tribological Properties

Wear Performance of the CNTs/Ceramics THA

Several research groups have conducted studies of CNTs / ceramic composites' tribological performances on thin (coating) and bulk forms. An investigation of the lubrication nature of CNT/ceramic composites conducted by Xia et al. showed that the contact and buckling behavior of the CNTs was influencing the friction coefficient of the composites [60]. Other investigations on CNT/ceramic composites thin coating revealed that the presence of CNT

up to 20 wt% of CNT exhibited improve wear resistance with lower friction with the increase of weight percent [61]. Therefore, CNT reinforced ceramic coating may be capable withstand high load-bearing conditions. These composites will be suitable for biomedical implant applications, such as total hip and knee arthroplasty.

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SEM microstructure for the microstructure of CNT/ceramic composites, as shown in Figure 14 (a), tends to agglomerate into a rope or bundle-like shape. CNTs have in composites have relatively high surface areas, high aspect ratio, and usually interact less with either solvents or matrix components [59]. SWCNT, in particular, consists of many parallel nanotubes held together by van der Waals forces [62].

The worn surface, shown in Figure 14b, is of CNT/composites suffering under the applied load, producing grooves and ridges pattern parallel to each other in the slide direction. Grooves and ridges exist when materials' surfaces deformation and micro-plowing occurs during a sliding contact between two contacting materials.

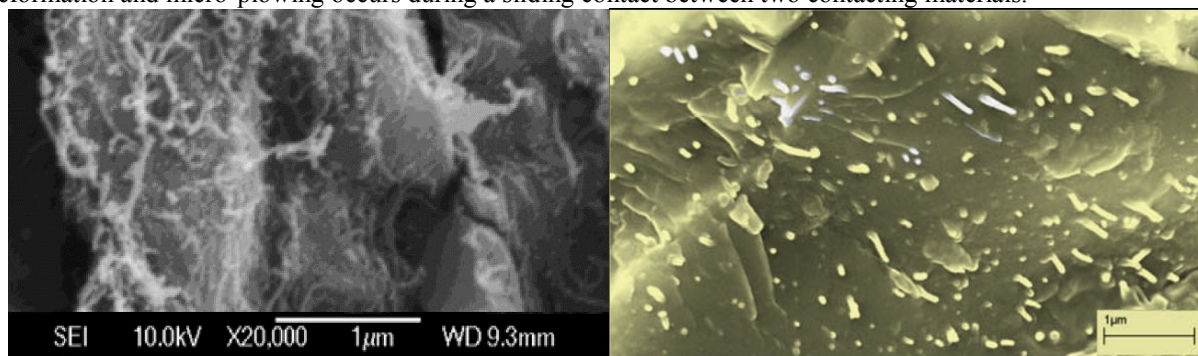


Figure 14:- (a) a typical SEM micrograph for CNT/Ceramic composites microstructure [63]; (b) Worn CNT/ceramic surface of CNT/ceramics [64].

It has been indicated from the literatures reviewed that research focusing on the wear mechanisms and wear performance of CNT/ceramics is still in the early step [41, 42, 63]. Simple studies about mechanical properties reported that there was no clear proof connecting between the quantitative performance data and the actual mechanisms. In the case of wear performance, the pull out of CNT during a sliding contact indicates the presence of energy-dissipation of interfacial friction; however, Scanning Electron Microscopy (SEM) images typically show relatively few CNTs emerging from the composite fracture surfaces [41, 42, 60, 63]. It is required, further work relating properties to wear mechanisms to consider how wear mechanisms may measure as the fiber diameter is in the nanoscale.

Plastic deformation, cracking and chemical reaction are the critical process involved in wear mechanisms in the wear of ceramics. Furthermore, the microstructures, such as grain size and porosity take a key role in indicating the ceramics wear properties and wear mechanisms. Therefore, understanding the wear mechanism of ceramic-on-ceramic hip prostheses requires research on material microstructures. However, research works on ceramic-on-ceramic hip prostheses have concentrated on wear performance, such as the wear rate and wear debris generation of the components rather than on the wear mechanisms [65, 66].

Wear Rate of CNTs/Ceramic THA

Some authors investigated the friction and wear behavior of the CNT composites under lubricated and unlubricated conditions. The wear rate decreased with increasing the CNT up to a small amount of 4 wt.% but rise with a further CNT addition. However, other preparation methods showed the wear-rate, decreased steadily with increasing CNT addition up to 12 wt.% [67]. Other authors reported that Tetragonal zirconia doped with three mol % of Ytria (3Y-TZP) has outstanding mechanical properties: high hardness, strength, and fracture toughness [68, 69].

Lots of efforts to create semi-quantitative models for the Wear Rate - Grain Size (W-G) relationship have used the Hall-Petch equation. As a result of the calculation of the equation above, it is shown in Figure 15 below that the

wear process involves damage to the sub-grain dimensional scale is assumed to be able to determine the macroscopic strength or the size of the critical damage.

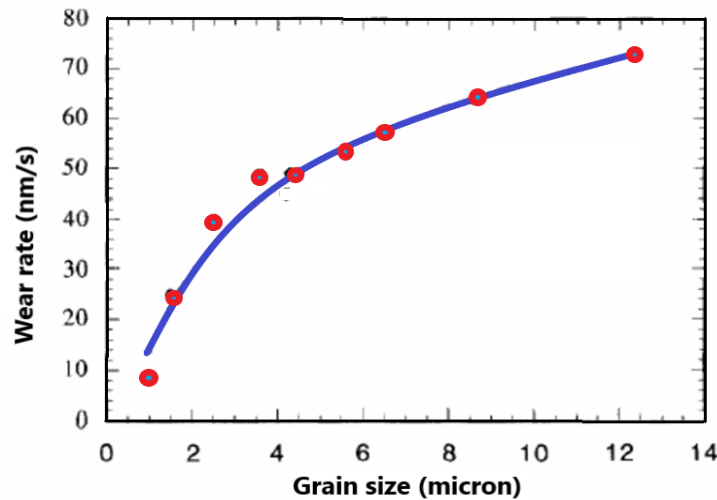


Figure 14:- Typical of the wear rate vs. grain size relationship in the lubricated pure ceramics [70].

As seen from Figure 15 for wet erosion, the wear rate (W) is a function of grain size (G) is not linear, but the relationship takes the form of a simple parabola. The wear process of single-crystal material is much slower than expected from extrapolation G to infinity. The concentrated high grain boundary presence of fine-grained provides better wear resistance than single-crystal materials.

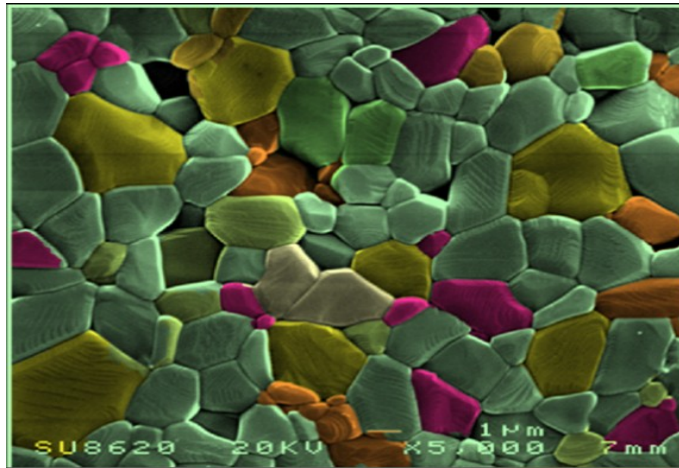


Figure 15:- Grain shape and size of the ceramics in micron-scale [71]

Figure 16 shows the poor microstructure, inadequate purity, low density, and rough grain contrasted to standard alumina for hip prostheses; these are considered as the key factors for the failure of THA ceramic on ceramic. In terms of the mean ceramic grain diameter ratio, the monolithic ceramic microstructure is different from the CNT-ceramic composite (Figure 14a).

Conclusion:-

Discussion of related ceramic materials used for Total Hip Arthroplasty (THA) femoral heads and cup liners components and reasons for the drawback and failure of conventional bioceramics for implants have been observed from the literature. The study of ceramic materials with carbon nanotubes (CNTs) in addition to conventional ceramics aimed to seek such new and advanced materials for implants. This paper also discusses the preparation technique for CNT-reinforced ceramic composites. The next step after this review is to prepare composite material research by adding CNT to conventional ceramic raw materials.

- (a) Published works have reported carbon nanotubes (CNTs) addition into pure ceramics could increase the ceramic material fracture toughness. Here are some important points regarding CNT's capability to strengthen ceramic materials:
1. The SPS is considered as a better method in making CNT ceramic composite. Observation has proven that almost double improvement of stiffness between hot pressed and SPS technique (from 100 to 300 MPa).
 2. Homogeneity of CNT dispersion is essential for obtaining significant properties improvement from the addition of CNT by 173% to 9.7 MPa^{1/2}.
 3. CNT has to be adequately blended to the matrix which serve as a medium of load transfer throughout the fracturing process.
 4. SWNT provides a better quality in the ceramic composite toughness than the MWNT. MWNT creates only slight interfacial strength, resulting in a point of failure during fracturing.
 5. CNT toughening mechanisms on the ceramic composite are followed by crack deflection, crack bridging, CNT ceramic interfacial strength, and fiber pullout.
- (b) The behavior of CNT's contact and buckling defines the composite frictional coefficient. The presence of CNT up to 20 wt% of CNT exhibited improve wear resistance with lower friction with the increase of weight percent. CNT reinforced ceramic coating may be capable to withstand high load-bearing conditions. These composites will be suitable for biomedical implant applications, such as total hip and knee arthroplasty.
- (c) Regarding wear performance, the pull out of CNT during a sliding contact indicates the energy-dissipation presence of interfacial friction. It is required, further work relating properties to wear mechanisms to consider how wear mechanisms may measure as the fiber diameter is in the nanoscale.
- (d) Granular pulling will also occur in ceramic prostheses, but the surface wettability is better than the bearing materials of the conventional Metal on Polymer (MoP). Wear particle generation during bearing surfaces sliding-contact could lead to an increase in surface roughness subsequently leading to a higher wear rate.
- (e) Deformation of plastic, cracking and chemical reaction are the critical process involved in wear mechanisms in the wear of ceramics. The microstructures, such as grain size and porosity take a key role in indicating the ceramics wear properties and wear mechanisms. Understanding the wear mechanism of ceramic-on-ceramic hip prostheses requires research on material microstructures.
- (f) The scar diameter increases with sliding time elapse. The deformation controlled wear scar diameter and free from grain size effect.

Acknowledgments:-

The authors would like to thank the Ministry of Research and Technology/National Agency for Research and Innovation and the LPDP for their funding support in the national research program schema. Likewise, the authors would like to thank BPPT for all support and approving the implementation of THA's materials technology development activities at the Materials Technology Center (PTM). All authors contributed equally to this work.

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