Preparation of nano-biocompatible ceramic composite

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Abstract

Nano – biocompatible ceramic composite material have been prepared ,this material consist of Alumina reinforced by nano cubic zirconia stabilized using magnesia, the idea of using MgO as a stabilizer is to utilize it as a sintering aid ,also to be comfortable in using the stabilization percent because we used a higher percentage than the percentage required to obtain a completely stable "cubic" phase, the remaining quantity of magnesia will form spinel which is a biocompatible ceramic material.XRD results proved the formation of $MgAl_2O_4$. TEM result assured the nanosize of the synthesized cubic zirconia ,which also assured using sherrer's equation.SEM was used to study the microstructure of the composite .

Introduction

Ceramics are brittle materials as they possess high mechanical properties but have low fracture toughness ,but if the nanoscale concept is used to create structures and then use those nanostructures to construct larger materials, nearly any set of properties can be obtained, create materials by design."[1] alumina is one of the most widely used ceramics because of its excellent biocompatibility ,high hardness and wear resistance ,[2]mono-phase bioceramic (Al_2O_3 and ZrO_2) are widely used in THR as an alternative to metal devices [3], the first suggestion of the application of Al_2O_3 ceramics in medicine came in 1932 but the bioceramic field did not develop until the 1970s with the first hip implants using Al_2O_3 balls and cup [4][5][6] unfortunately ,the orthopaedic community reports significant in-vivo failures[3] the resistance of brittle materials to the propagation of cracks can be strongly influenced by microstructure and by the use of various reinforcement [7],. Alumina and zirconia are bioinert ceramics; their most popular application is in arthroprosthetic joints where they have proven to be very effective, that make their use suitable especially in younger, more active patients. Also dental use of these materials was proposed to achieve aesthetic and reliability of dental restorations[8]. In biomedical applications, alumina has considerable advantages over other materials because of its inertness, which offers excellent biocompatibility and nonsensitization of tissues. [9] Zirconia alumina is a mixture of zirconium dioxide and aluminium oxide. Zirconia toughened alumina with a 10 to 20 percent zirconia concentration, which enhances the strength of the alumina.[10] AZ composites are commonly used in structural applications, as cutting tools, and in many medical applications. Additionally, AZ composites feature high strength, fracture toughness, elasticity, hardness, and wear resistance.[11] Zirconia alumina is also used in the medical industry, primarily in joint replacement parts. The wear resistance, durability and ability to withstand

great weight make zirconia alumina the material of choice for orthopedic implants. In addition, Cut-off wheels made from zirconia alumina are preferred for pre-finishing many types of implants, because they cut faster than wheels made of alumina only, last many times longer, and work well on the hardened materials used in implants.[12]spinel is used as abiomaterial [13]14][15][16],

The formation of spinel during the sintering of alumina using magnesia as a sintering aid suppress the grain growth[17]In-Ceram Spinel as its major crystalline phase contains a magnesium spinel (MgAl2O4) but retains traces of alpha-alumina. The spinel improves the translucency of the core and final restoration[18]

Experimental procedures

Nano Cubic Zirconia powder has been prepared by precipitation technique by using NH_4OH as the precipitant ,in this method we followed the following steps:

1) Weighing the appropriate amounts of $ZrOCl_2.8H_2O$ and $MgCl_2.6H_2O$. (20mol% MgO +80 mol% ZrO_2)
2) Dissolving the chlorides in distilled water to form a stable chlorides solution.
3) Adding the chlorides solution to NH_4OH under vigorous stirring.
4) Aging of the precipitates for 15minutes to accomplish the completation of the precipitation .
5) Washing of the precipitates by hot distilled water in the beaker until the CL^{-1} ions are no longer exist within the gel ,this is achieved using the PH meter and $AgNO_3$.
6) Washing of the precipitates by ethanol " in the beaker and in the centrifuge ".
7) Filtering .
8) Drying at 120°C.
9) Calcination at 800°C.

This procedure have been concluded from the previous works and our understanding to the whole concept of chemical precipitation ,we didn't depend on the previous works only because of the difference in the materials used and the phase required. After that XRD test was conducted to identify the material, and the particle size was calculated using sherrer's equation ,

$$\tau = \frac{k\,\lambda}{B\cos\theta}$$

Where:τ is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size;K is a dimensionless shape factor, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite ;λ is the X-ray wavelength ;β is the line broadening at half the maximum intensity (FWHM), after subtracting the instrumental line broadening, in radians. This quantity is also sometimes denoted as $\Delta(2\theta)$;θ is the Bragg angle,

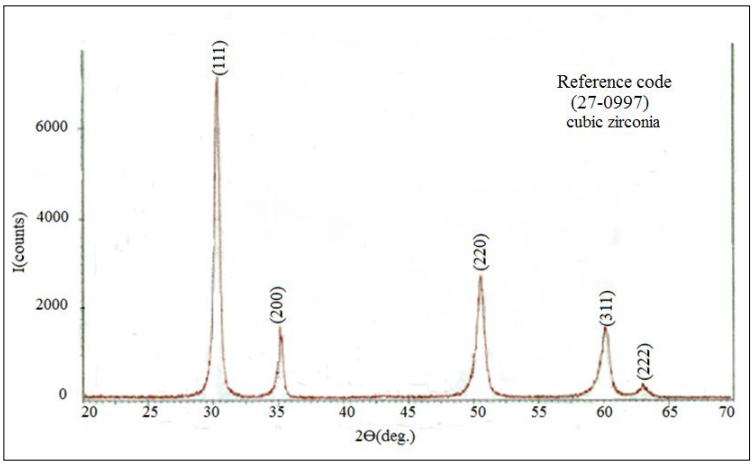
measured by particle size analyzer, and TEM. Pure alumina powder was mixed with the precursor of the nano cubic zirconia at different contents (0%,3%,5%,10%,and 15% zirconia precursor) . The samples were prepared by uniaxial pressing at 150MPa using steel moulds ,heat these samples were finally sintered at 1500 °C for 2 h in air with a heating rate of 10 °C/min. Bending strength was measured using the universal testing machine.

XRD ,SEM ,EDS tests were used to examine the samples .

Results and discussion

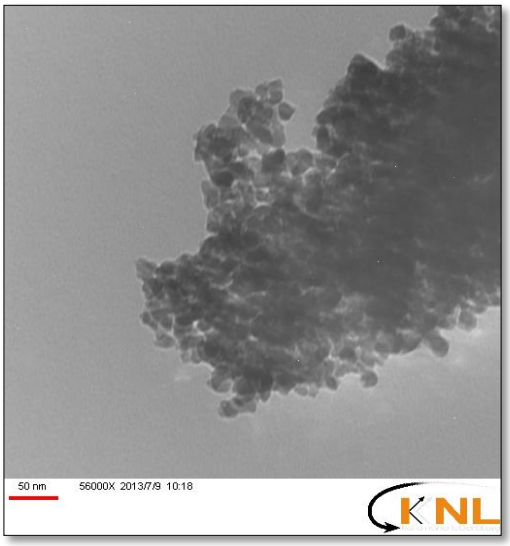
Particle size of zirconia

The particle size of the synthesized Zirconia powder was calculated using Scherrer equation,The wave length of the X-Ray beam =1.5406Å and from the chart β=0.034 radian, θ= 15.25 degree ,these value were put in the above equation and the crystallite size was found to be 42.26Å.The XRD chart used to evaluate the apply this equation is shown below in fig.(1)



Figure(1):XRD chart of the synthesized nano-zirconia

Figure(2) shows the images of the TEM results ,and these images are the most specified technique used to estimate the particle size, from these images it is obvious that the particle size is in the nano range.



Figure(2):TEM image of the synthesized nano-zirconia.

XRD of the composite :

The following chart shows the XRD results of the samples after sintering:

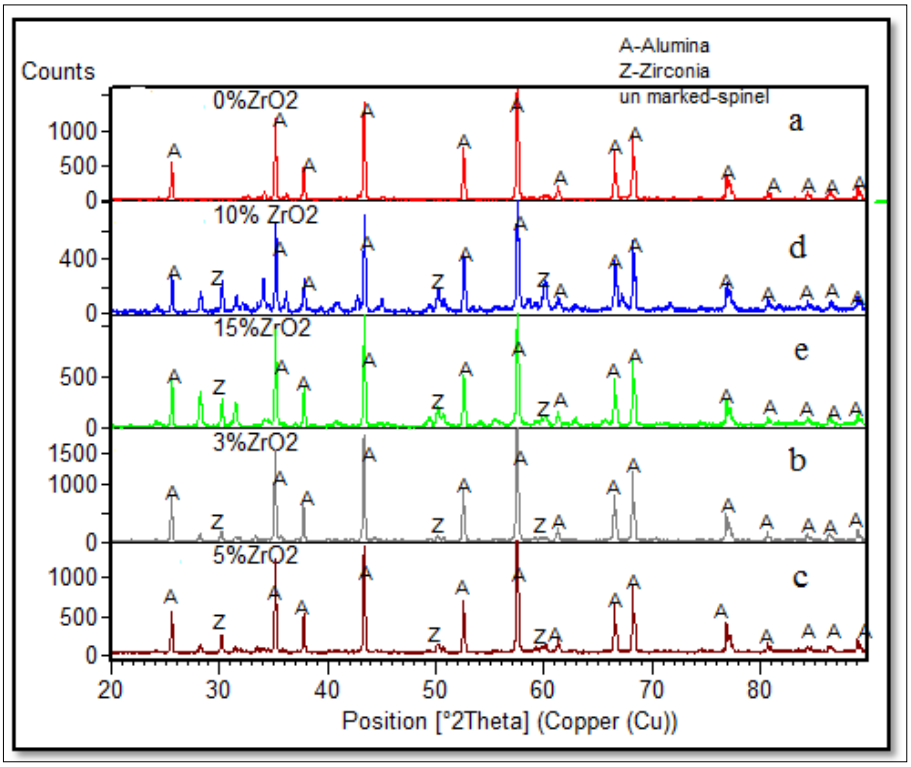


Fig.(3):XRD charts of the samples after sintering.

The X-Ray analysis shows that the synthesized nano zirconia consists of fully cubic phase fig.(1)X-Ray is also used to examine the alumina and alumina containing different fractions of ZrO_2.Figure (a) shows the spectra for the alumina sample which is completely α-Al_2O_3 ,while figures (b),(c),(d), and (e) showing the spectra for the ceramics of alumina with different zirconia content (3 wt%,5 wt%,10 wt% ,and 15 wt%) respectively, after sintering at 1500°C .The results indicates the presence of the cubic phase co-exist with α-Al_2O_3matrix and also indicates the formation of the spinal structure $MgAl_2O_4$,from the spectra of theAl_2O_3-ZrO_2 the intensity of the spinal structure increase with the increment of zirconia content ,and ZrO_2 remain cubic after sintering" this indicates that the material is very stable" The formation of $MgAl_2O_4$ resulted from the following reasons :

1- The nanosize of Mg-stabilized zirconia.

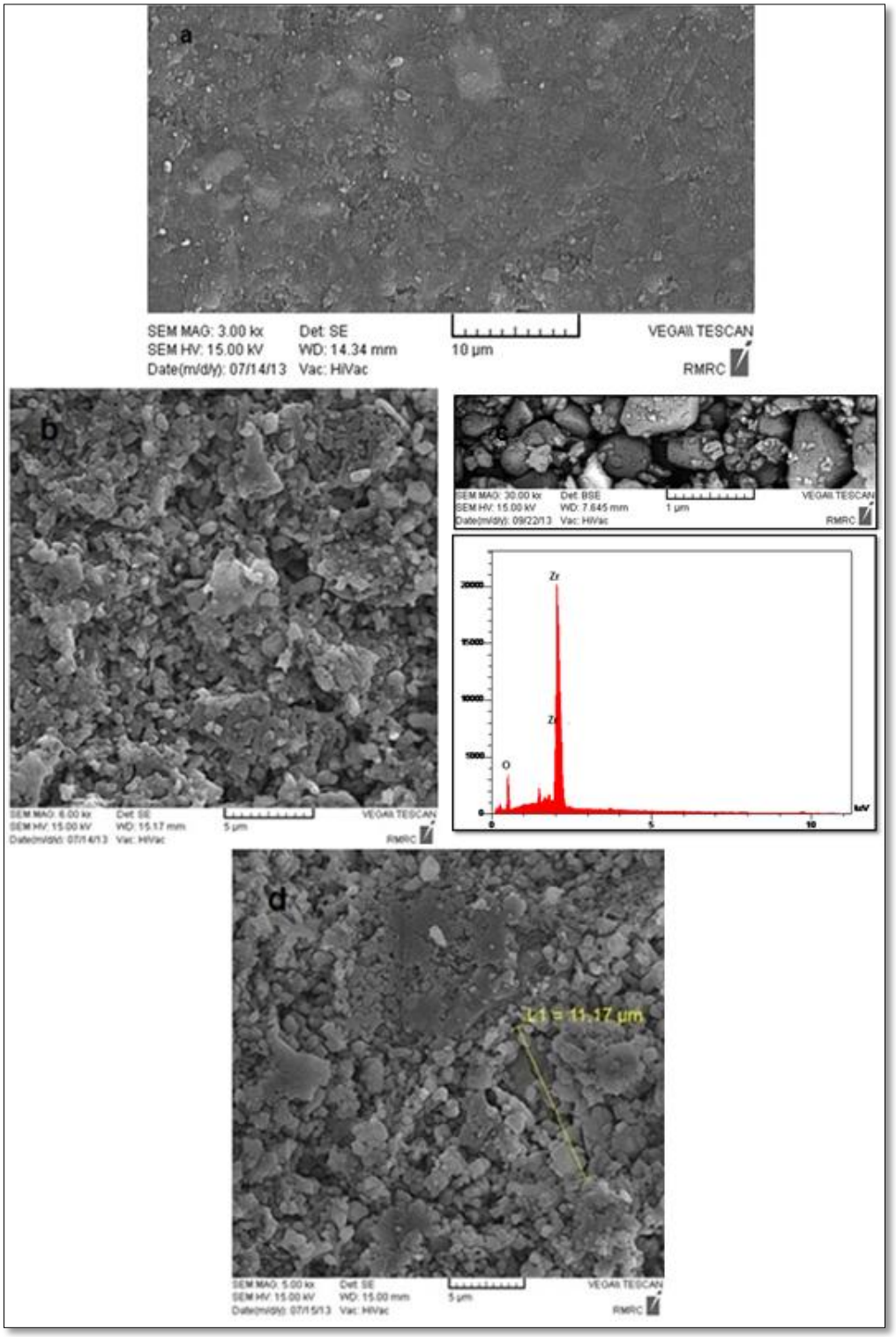
2-The percentage of MgO used to stabilize zirconia in the cubic phase at room temperature.

3-The method used to disperse zirconia within the alumina matrix.Since the particle size of zirconia is in the nano range this leads to higher surface area of the MgO particles which means high reaction rate ,also the content of MgO used was higher than the required amount but it was selected based on the phase diagram of ZrO_2-MgO to ensure the formation of the cubic phase. Also the method used to prepare the composite material's powder helped to form the spinal structure since it was not traditional " mixing the hydrated zirconia with alumina in ethanol using ultrasonic waves and then mixing using magnetic stirrer until the complete evaporation of ethanol " which allows homogeneous

distribution of the nano Mg-stabilized zirconia within the alumina matrix .

4- The presence of zirconia increase the solubility "this was claimed by Charles *et al*. in their patent on the addition of ZrO_2+ MgO addition to Al_2O_3, Zr^{+4} charge compensate for the Mg^{+2} in the Al_2O_3 lattice and increases the solubility.[7],[19]

SEM and EDS :



Figure(4):SEM images EDS of the samples

To study the microstructure and the fracture surface of the Al_2O_3-ZrO_2 ceramics .SEM investigations into the crack path indicate that some cracks are impeded and deflected , and microcracking . Figure (4-a):image of the surface of the 15%ZrO_2 sample. shows SEM image of the fracture surface of samples of 5%ZrO_2 sample. The EDS result for the bright spots is shown in the fig.(4-c) and it obvious from the chart that they are ZrO_2, the same figure shows SEM image of 3% ZrO_2 used in the EDS test,while figure (4-d)shows SEM image of the fracture surface of samples of 5%ZrO_2 sample.

Conclusions

1. Chemical precipitation is an effective method to synthesize nano-cubic zirconia.
2. Spinel can formed using this procedure at relatively low temperature compared with the conventional methods.
3. A strong bioceramic (alumina /nano-cubic zirconia) can be prepared utilizing the nanoscale effect.

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