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Effect of zirconia (ZrO₂) nanoparticle addition on (dimensional change, compressive strength, creep, and vickers micro hardness) of the high copper dental amalgam

ABSTRACT

This research is aimed at understanding the effects of one of the components of dental amalgam: the mechanical and structural characteristics of dental amalgams caused by nano-sized Zirconia Ceramics. Some of the mechanical tests done were Vickers hardness, creep resistance, compressive strength, and dimensional change and some microstructural tests were done using x-ray diffraction and optical microscopy. These tests were done with the aid of amalgam that contains Zirconia Ceramics and the amalgam that contains 0–3% of Zirconia Ceramics.

Around 2% ZrO₂, Creep resistance increased by 44% as the percentage of creep resistance increased from 1.29% to 0.722%. An improvement of 18.5% in dimensional stability was also observed, as 3% ZrO₂ resulted in a decrement of 0.135% to 0.11%. ZrO₂ resulted in a 14.9% improvement, from control values of 328 MPa, and a ZrO₂ addition of 3% increased compressive strength to 377 MPa. Vickers hardness increased by approximately 54% from control values and also increased with the 2% addition to 200 HV. Furthermore, the 2% addition produced the greatest Vickers hardness.

XRD confirmed a composite of Zirconia, and also of Ag₂Hg₃, and Cu₆Sn₅ phases, which was formed from the metallic and ceramic powder components. Nano Zirconia additions suggest substantial improvement to dental amalgams with regard to mechanical and deferred stability, and improvement towards clinical applications.

Keywords: Amalgam, ZrO₂, admix, creep, compressive strength, dimensional change

1. INTRODUCTION

For the rest of the dental materials a lot of research is incorporated with a greater dependence on the manipulation of materials and the characteristics of the dental materials. Out of all of these materials, dental amalgam stands out the most [1,2].

It makes up about 75% of all restorative dental materials used [3,4]. Introduced over 150 years ago, dental Amalgam is an alloy of metal that has been modified or used as the main and efficient restorative material in dentistry. Amalgam is hypothetically called "silver amalgam," but really, it is a mixture of several metals. It consists of silver, mercury, tin, and copper. Amalgam can also have very small quantities of zinc, indium, or palladium.

However, in recent years, its accolades as a restorative material have decreased considering the fears of negative health effects and environmental pollution [5,6].

Amalgams can be classified into low copper and high copper. While all amalgams tends to corrode in the mouth, high copper amalgams have been known to corrode less. [7]. Although its use is rapidly declining around the world, Amalgam has retained the preference of many dentists as the method or material of choice to replace posterior teeth as it is considered to be economical, durable, and easy to manipulate. However, it is very important to be careful of the corresponding diagnosis [8]. Amalgam can be synthesized by mercury and an amalgam alloy and it should be the amalgam alloy that is mixed into the mercury [9, 10].

There is little effect on the environment when amalgam is in the environment. It has been shown that amalgam is a low contributor to human exposure from soil or from an inhalation exposure path.

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Yet, the Scientific Committee on Health and Environmental Risks (SCHER) argues that no scientific data validates these assertions at present. Therefore, it is suggested that other materials for dental amalgams may be investigated [11,12].

Hence, the purpose of this study is to analyze the effect of nano zirconia reinforcement on the properties of dental amalgam filling.

Dental amalgam has been researched and perfected for years, however, there are still issues with the long-term aging of this material, mainly its mechanical stability and its dimension changes. Studies done in the past have attempted to add different types of nano sized additives/ceramics to restorative materials in dentistry. Not many have tried to add nano zirconia (ZrO₂) to mixed dental amalgam systems and to see how that correlates with the mechanical properties of creep resistance, compressive strength, dimension change and hardness.

Also, in many previous studies, the focus was more on the evaluation of a single property or macro-scale additives. The potential of the nano-scale ceramic particles and the impact of the microstructural morphogenesis of amalgam matrices are still not addressed in sufficient detail. Additionally, few studies have examined both the mechanical properties and the microstructure of nano-reinforced dental amalgam.

Consequently, there is a pronounced requirement for a methodical study on the influence of ZrO₂ at the nano level on the dental amalgam. This study will try to ensure this gap is filled through the assessment of several different percentages of ZrO₂ nano particles and their effect on the compressive strength and hardness, stability in dimensions.

2. MATERIALS AND METHODS

Amalgam Specimen Preparation

The specimens were manufactured in accordance with ADA specification No. 1 [13] through the trituration of alloy powder and mercury at a 1/0.9 ratio. Amalgams consist of mercury with a purity of 99.99% and a powder composed of 40% Ag, 31.3% Sn, and 28.7% Cu, combined with a ceramic addition of nano zirconium oxide with particle sizes ranging from 10 to 20 nm, the addition (0, 1, 2 and 3 wt %), processed using a mechanical amalgamator for thirty seconds. The specimens are four millimeters in diameter and eight millimeters in height. A conventional condenser with condensing point of two millimeters diameter was utilized to physically pack the mixture into a Teflon mold of suitable size, according to

the ADA, positioned on a horizontal surface. The specimens of each addition were prepared and aged at 37±1°C for test time prior to testing on a horizontal surface [14].

Microstructure Observation

Optical microscopy was employed to characterize the amalgam morphology. This test was conducted in the Materials Department at the University of Kufa using the NMM-800RF from Xiamen Phio, China.

The amalgam specimen is cold mounted with polyester resin that is cured at room temperature to facilitate microstructural observation. The samples were ground and polished with diamond paste of particle size 0.25 µm and progressively finer grades of emery paper, ranging from 220 to 3000. The specimens were meticulously cleaned with distilled water and dried with an air blast. A 30% nitric acid etchant was employed to etch the polished amalgam alloy specimens. A 400x magnification optical microscope was subsequently employed to analyze the samples.

X-ray diffraction was conducted to ascertain the crystallographic phase characteristics of the amalgam. The test was conducted at the University of Kufa's Nanotechnology and Advanced Materials Research Centre utilising XRD (LabX 6000, Shimadzu, Japan) equipped with a nickel filter and a copper K α radiation generator ($\lambda=0.154056$ nm).

Dimensional Change

The initial measurement for dimensional change for five specimens of each type was conducted 5 minutes post-trituration, utilizing a digital micrometer with an accuracy of 0.01 µm to assess the specimen's length. Twenty-four hours after the trituration was completed, the specimens' length was measured again. Throughout the examination, the specimens were maintained at a temperature of 37± 1°C according to the A.D.A. specification No. 1 stipulates that the dimensional variation must range from -0.15 to +20 µm/cm for acceptance. The percentage of dimensional change is determined by dividing the change in length observed between five minutes and 24 hours post-trituration by the original length, Equation 1 computes dimensional change as follows:

$$\text{Dimensional change Percent \%} = \frac{\Delta L}{L_0} \times 100 \quad (1)$$

Where:

L_0 = length after five minutes

ΔL = change in length after 24h.

Compressive Strength

Compressive strength tests were conducted for five specimens of each type using a universal testing machine of type WDW 200, which was made in China. For testing, a constant loading speed of 0.5 mm/min was employed. The first measurement was obtained an hour after the trituration was finished, and the second measurement was done 24 hours later. These specimens were kept at a constant temperature of 37±1°C until the experiments were finished. Equation 2 computes compressive strength (σ) as follows :

$$\sigma = \frac{F}{A} \quad (2)$$

Where:

σ :Compressive strength in Mpa is represented.

A: Cross sectional area in millimeters

F: Maximum force in N

Creep

A creep test, also known as a stress-relaxation test, assesses the extent of deformation a material undergoes over time when subjected to a continuous compressive load at a constant temperature [15].

Before performing the test, emery paper was employed to smooth both sides of three specimens of each group type. It is essential to measure the specimen length prior to the application of the load. The specimens were exposed to an axial tension of 38 MN/m² for four hours at a constant temperature of 37 ± 1 °C. The specimen length was measured after four hours of load application, calculated as follows: the change in length between 1 and 4

hours of the specimen under the applied load divided by the original length yields the creep percentage Equation 3 computes Creep Percent as follows [16].

$$\text{Creep Percent \%} = \frac{L_o - L}{L_o} \times 100 \quad (3)$$

Where:

L_o = original length,

L = final length.

Vickers hardness

Vickers' microhardness was conducted using a digital micro-hardness tester (TH715, Time High Technology Ltd, China). It is equipped with a 400X magnification microscopic lenses. The hardness readings were taken in three different locations by using a 9.8 N load with dwell time 15 sec for three specimens of each type; the average of these three readings was determined for each sample of all the addition groups.

3. RESULTS AND DISCUSSION

Microstructure Observation

Figure 1 illustrates the microstructure of dental amalgam with 3% ZrO₂ resulting from the etching with 30% nitric acid within the amalgam microstructure. The dark-hued unreacted particles are encased within a continuous γ_1 (Ag₂Hg₃) matrix and η (Cu₆Sn₅) phase.

The matrix of the γ_1 phase exhibits a lighter color. Under microscopic examination, the spaces appear as dark were the unreacted particles. Thus, the final microstructure of the amalgam mass consists mainly of unreacted particles encased in a matrix of reaction products. The unreacted particles are retained by the matrix.

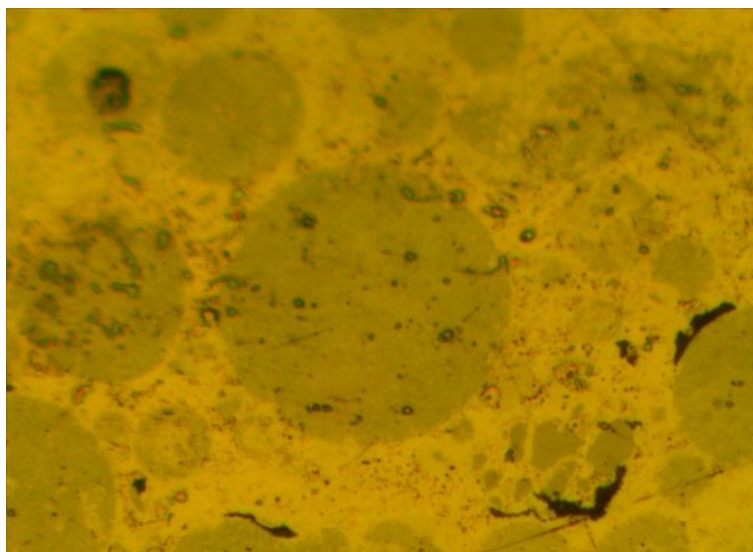


Figure 1. Illustrates microstructure of dental amalgam with 3% ZrO₂

The XRD pattern confirmed the presence of the major crystalline phases of the high-copper dental amalgam, including γ_1 (Ag_2Hg_3) and η (Cu_6Sn_5), together with diffraction peaks corresponding to the incorporated ZrO_2 nanoparticles [17,18]. The identified phases are indicated by blue squares (Ag_2Hg_3), red circles (Cu_6Sn_5), and black inverted triangles (ZrO_2), as shown in Figure 2. The diffraction pattern exhibits several characteristic reflections over the 2θ range of 20° – 80° , with the highest peak density observed between 30° – 45° and 60° – 75° . It should be noted that the diffraction feature around $2\theta \approx 38^\circ$ was not assigned exclusively to ZrO_2 because this region may

involve overlapping reflections from the amalgam phases, particularly γ_1 (Ag_2Hg_3), in addition to the incorporated zirconia nanoparticles. Therefore, the identification of ZrO_2 was based on the overall diffraction pattern and its characteristic reflections rather than on a single diffraction peak.

The coexistence of γ_1 (Ag_2Hg_3), η (Cu_6Sn_5), and ZrO_2 confirms the retention of the characteristic amalgam phases after the incorporation of zirconia nanoparticles, indicating that the added ceramic phase was successfully incorporated without altering the primary crystalline constituents of the amalgam.

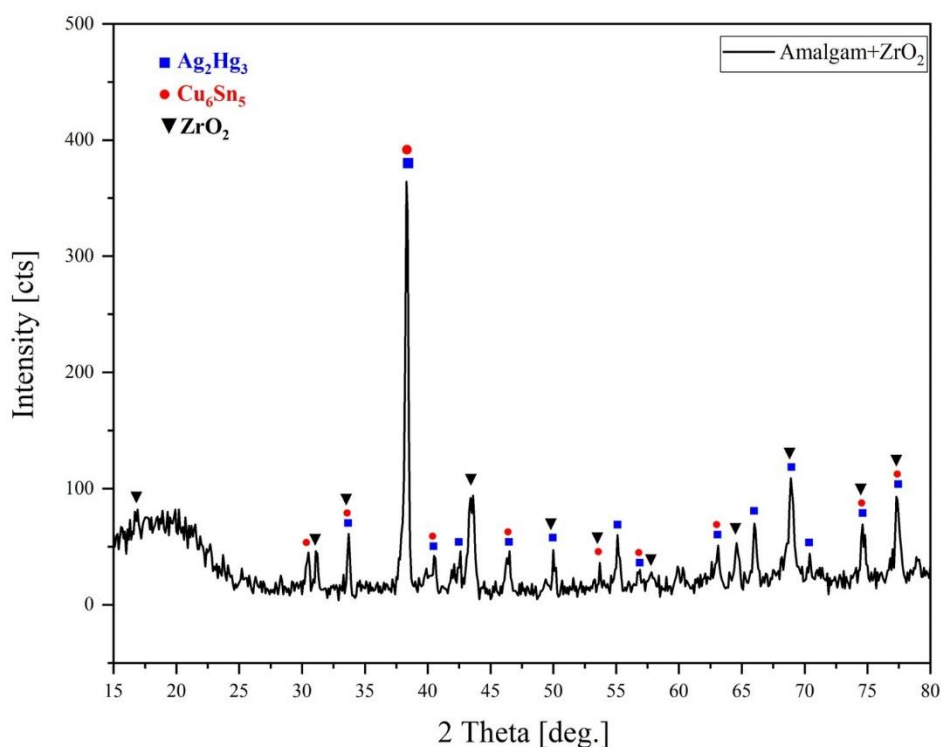


Figure 2. XRD pattern of dental amalgam alloy with 3% ZrO_2

Dimensional Change

Table 1 illustrates the correlation between ZrO_2 and the percent Dimensional Change, suggesting that an increase in Addition percent results in a decrease in Dimensional Change. With a 0% Addition control specimen, the dimensional change is about 0.14%. When the Addition percent is about 3%, the change is reduced to about 0.12%. The dimensional change continues to decrease with an increase in ZrO_2 addition. The improved dimensional stability can be attributed to the nano-sized ZrO_2 particles filling the interparticle spaces within the amalgam matrix, resulting in a denser structure and reducing volumetric changes during the setting reaction [19].

Table 1. The Mean Value and Standard Deviation of Dimensional Change Test.

$\text{ZrO}_2\%$	Dimensional Change	S.D.	Improvement %
0	0.135	0.01	0
1	0.127	0.014	5.9
2	0.115	0.011	14.8
3	0.11	0.016	18.5

Compressive Strength

Table 2 presents the effect of ZrO_2 addition on the compressive strength of dental amalgam at 1 h and 24 h of aging. At 1 h, strength increased from

82 ± 3.3 MPa (control) to 94 ± 3.2 MPa at 3% addition. At 24 h, strength increased from 328 ± 8.1 MPa (control) to 377 ± 7.41 MPa at 3% addition, a 12.9% improvement. The increase in compressive strength is attributed to the reinforcing effect of

ZrO₂ nanoparticles. Their high hardness and elastic modulus restrict localized plastic deformation, while their uniform distribution improves load transfer within the amalgam matrix, thereby enhancing its load-bearing capacity [19, 20].

Table 2. The Mean Value and Standard Deviation of the Compression Strength

ZrO ₂ %	Compressive Strength at 1h	S.D.	Improvement %	Compressive Strength at 24h	S.D.	Improvement %
0	82	3.3	0	328	8.1	0
1	86	3.24	4.9	335	11.2	2.1
2	90	5.2	9.8	374	9.08	14.0
3	94	3.2	14.6	377	7.41	14.9

Creep

Table 3 illustrates the relationship between "Creep %" and "ZrO₂ Addition %". At the control specimen (0% Addition), the creep percentage is roughly 1.3%. Once the addition percentage reaches roughly 2%, the creep percentage has been noted to decrease to its lowest. Here, the rate of creep percentage is almost 0.7%. After an increase to 3%, the creep percentage is observed to begin to increase. Beyond 2%, creep resistance may not improve. To 2%, the addition may improve the material's creep resistance. The addition may improve, to some extent, the material's worth, but over, let's say, 2%, the inclusion may lessen the material's value, or the effect may even be slightly negative. Thus, the recommendation to minimize creep is to set the addition to 2%. The improved creep resistance is attributed to the rigid ZrO₂ nanoparticles, which restrict time-dependent deformation of the amalgam matrix under sustained loading. The slight increase in creep at 3 wt.% may be associated with partial nanoparticle agglomeration, reducing the reinforcing efficiency [20, 21]. The highest improvement in creep resistance (44.0%) was achieved at 2 wt.% ZrO₂, whereas a slight reduction was observed at 3 wt.%, which may be attributed to partial nanoparticle agglomeration.

Table 3. The Mean Value and Standard Deviation of the Creep Test

ZrO ₂ %	Creep %	S.D.	Improvement %
0	1.29	0.02	0
1	0.925	0.1	28.3
2	0.722	0.09	44.0
3	0.742	0.11	42.5

Vickers hardness

Table 4 illustrates the correlation between the percentage of ZrO₂ and Vickers Hardness. As the

ZrO₂ percentage rises from 0% control specimen to around 2%, the hardness consistently escalates. Beyond around 2%, the hardness commences stabilizes. The hardness attains its zenith at around 3% addition, with a peak Vickers hardness slightly exceeding 200. The preliminary rise in hardness indicates that the addition enhances the material's hardness up to a specific concentration. Exceeding the optimal threshold may result in saturation or adverse consequences such as brittleness, phase segregation, or diminished bonding, which marginally decreases hardness. A precise addition of 2% produces the maximum Vickers' hardness. The increase in hardness is mainly attributed to the intrinsic hardness of ZrO₂ nanoparticles and their uniform dispersion within the amalgam matrix, which enhances the resistance to indentation and limits localized plastic deformation [21].

Table 4 The Mean Value and Standard Deviation of the Vickers hardness

ZrO ₂ %	Vickers hardness	S.D.	Improvement %
0	133	3.15	0
1	174	9.3	30.8
2	205	9.2	54.1
3	206	8.8	54.9

4. CONCLUSION

The use of nano-sized zirconia (ZrO₂) particles within dental amalgam provides a remarkable improvement in the amalgam's mechanical and physical properties. Experimental data revealed that, with the appropriate addition of ZrO₂, significant enhancements were achieved in creep resistance, dimensional stability, compressive strength, and hardness. The creep resistance exhibited a maximum improvement of approximately 44% at 2 wt.% ZrO₂, where the creep value decreased from 1.29% to 0.72%.

Dimensional stability improved by about 18.5% at 3 wt.% ZrO₂, as the dimensional change decreased from 0.135% to 0.11%.

Furthermore, compressive strength showed a noticeable increase, reaching up to 377 MPa at 3 wt.% ZrO₂, corresponding to an improvement of approximately 14.9% compared to the control sample. In addition, Vickers hardness demonstrated a significant enhancement of about 54%, increasing from 133 HV for the control sample to over 200 HV at 3 wt.% ZrO₂.

Among the investigated concentrations, the lowest creep value (0.72%) and the highest hardness were obtained at 2 wt.% ZrO₂, while the minimum dimensional change and maximum compressive strength were achieved at 3 wt.% ZrO₂, indicating that the optimal performance depends on the targeted property.

Through the use of microstructural and XRD analysis, a matrix comprised of the composites Ag₂Hg₃, Cu₆Sn₅, and ZrO₂, containing metallic and ceramic phases, was observed to promote the proof of concept of ZrO₂'s positive effect as a supportive substance. These findings support the incorporation of ZrO₂ into future dental restorative material formulations. Further studies are warranted to evaluate the long-term clinical performance and biocompatibility of ZrO₂-reinforced amalgam within the oral cavity.

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IZVOD

UTICAJ DODAVANJA NANOČESTICA CIRKONIJUMA (ZrO₂) NA (PROMENU DIMENZIJA, ČVRSTOĆU NA PRITISAK, PUZANJE I VIKERSOVU MIKROTVRDOĆU) ZUBNOG AMALGAMA SA VISOKIM SADRŽAJEM BAKRA

Ovo istraživanje ima za cilj razumevanje efekata jedne od komponenti zubnog amalgama: mehaničkih i strukturnih karakteristika zubnih amalgama izazvanih nano-dimenzionalnom cirkonijumskom keramikom. Neki od mehaničkih testova koji su sprovedeni bili su tvrdoća po Vickersu, otpornost na puzanje, čvrstoća na pritisak i promena dimenzija, a neki mikrostrukturni testovi su sprovedeni pomoću rendgenske difrakcije i optičke mikroskopije. Ovi testovi su sprovedeni uz pomoć amalgama koji sadrži cirkonijumsku keramiku i amalgama koji sadrži 0–3% cirkonijumske keramike.

Oko 2% ZrO₂, otpornost na puzanje se povećala za 44%, jer se procenat otpornosti na puzanje povećao sa 1,29% na 0,722%. Takođe je primećeno poboljšanje dimenzionalne stabilnosti od 18,5%, jer je 3% ZrO₂ rezultiralo smanjenjem od 0,135% na 0,11%. ZrO₂ je rezultirao poboljšanjem od 14,9% u odnosu na kontrolne vrednosti od 328 MPa, a dodatak ZrO₂ od 3% povećao je čvrstoću na pritisak na 377 MPa. Vickersova tvrdoća se povećala za približno 54% u odnosu na kontrolne vrednosti, a takođe se povećala i sa dodatkom 2% na 200 HV. Štaviše, dodatak 2% je proizveo najveću Vickersovu tvrdoću.

XRD je potvrdio kompozit cirkonijuma, kao i faza Ag₂Hg₃ i Cu₆Sn₅, koji je formiran od metalnih i keramičkih komponenti praha. Dodaci nano cirkonijuma ukazuju na značajno poboljšanje zubnih amalgama u pogledu mehaničke i odložene stabilnosti, kao i na poboljšanje ka kliničkoj primeni.

Ključne reči: Amalgam, ZrO₂, dodatak, puzanje, čvrstoća na pritisak, promena dimenzija

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