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Effect of micro- Al_2O_3 addition on the compressive strength, creep, vickers microhardness, and thermal conductivity of high-copper dental amalgam

ABSTRACT

The main goal of this investigation is to study the effect of the (Al_2O_3) addition on (Compressive Strength, Vickers Micro hardness, Creep and Thermal conductivity) of the high copper dental amalgam. Casting was used to create one alloy made up of Ag (47 %), Sn (30 %), Cu (22 %), and Zn (1 %). (Al_2O_3) addition is made in various percentages (0.5, 1, 1.5, 2 wt%). ADA specification No. 1 was followed in the preparation of specimens. The specimens were stored at $37 \pm 1^\circ \text{C}$ in a glass chamber specially designed for this purpose. Compressive Strength of the amalgam increases with addition from 0.5% to 2% (Al_2O_3), creep of the amalgam decreases after addition from 0.5% to 2% (Al_2O_3) and micro hardness HV of the amalgam increases with increasing Al_2O_3 content. thermal conductivity of the amalgam decreases with addition from 0.5% to 2% (Al_2O_3).

Keywords: dental amalgam; Compressive Strength, Creep, Vickers Micro hardness; Al_2O_3 ; thermal conductivity.

1. INTRODUCTION

Teeth play a vital role in maintaining oral health and enhancing bodily appearance. Many individuals endure oral conditions such as caries and pulpitis. Specific periodontal ailments may compromise dental well-being and performance [1]. Dental amalgam restorations with mercury have a historical precedent dating back to the Chinese in 659 A.D. They were the initial users of silver-mercury past [2]. It has been utilized since the 1800s, typically with minimal documented adverse effects on humans [3]. Estimates indicate that 75% of all individual tooth restorations are comprised of amalgam. Moreover, this percentage stayed constant over various years. The ease of placement and manipulation, comparatively low cost, and established performance history in the oral cavity have rendered dental amalgams a highly popular restorative material. Dental amalgam is an alloy composed of liquid mercury. Additionally, it is composed of various metal alloy particulates, including silver, copper, and tin. Initially with the silver-mercury amalgams.

Subsequently, it was altered through the incorporation of tin to control a portion of the expansion. The amalgams produced a fragile, susceptible intermetallic γ_2 phase (Sn_7Hg). Consequently, a contemporary dental amalgam now incorporates copper (>12%) to inhibit this phase [5,6]. Amalgams are categorized into low copper and high copper varieties. Although all amalgams tend to erode in the oral cavity, high copper amalgams are recognized for exhibiting reduced corrosion. [7]. Despite its global decline in usage, amalgam remains the preferred choice for many dentists in replacing posterior teeth due to its cost-effectiveness, durability, and ease of manipulation. Nonetheless, it is crucial to exercise caution regarding the pertinent diagnosis [8]. The reaction between mercury and an alloy that occurs upon mixing is referred to as amalgamation. This reaction produces a hard restorative material of a silver-grey hue. The color restricts its applicability in voids where aesthetics are not a primary consideration [9]. The efficacy of amalgamation is significantly influenced by composition and kinetics; even minor additions can modify phase composition, microstructure, and degradation mechanisms, resulting in measurable impacts on mechanical properties and corrosion behavior [10]. Commercially pure titanium, characterized by an α -phase structure, exhibits significant corrosion

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resistance and is predominantly used in orthopedic and dental applications without inducing toxicity in the human body [11,12]. Amalgam fillings typically tarnish and exhibit discolouration, but they may also corrode on occasion. Tarnished amalgam restorations are unappealing and, if neglected, may result in deterioration. The oxidized layer primarily consists of tin sulfide, with sulfur originating from a sulfur-rich diet. Corrosion byproducts may infiltrate the dentinal tubules in the dentin, resulting in discolouration of the entire tooth. The corrosion products can seal the minor marginal gap, hence preventing marginal failure and leaking.

To avert tarnish and deterioration, cavity varnish must be applied to the cavity floor and walls prior to the insertion of restorative materials. Furthermore, all amalgam fillings must be meticulously polished and designed to be self-cleaning [13]. The environmental impact of amalgam is minimal. Amalgam has been demonstrated to be a minimal contributor to human exposure via soil or inhalation pathways. However, the Scientific Committee on Health and Environmental Risks contends that there is currently no scientific evidence to substantiate these claims. Consequently, it is recommended that alternative materials for dental amalgams be explored [14,15].

Moreover, numerous prior investigations have predominantly concentrated on assessing a singular attribute or the application of additives composed of alternative oxides. Therefore, a systematic investigation into the impact of aluminum oxide (Al_2O_3) on dental amalgam is urgently required. This work seeks to address the research gap by assessing the influence of different amounts of Al_2O_3 particles on compressive strength, hardness, creep, and thermal conductivity.

2. EXPERIMENTAL WORK

2.1. Preparation of the powder alloy

The basic alloy, comprising silver, copper, tin, and zinc, is initially melted in an electric furnace, with the procedure concluding entirely in an inert atmosphere utilizing argon gas. Casting was employed to fabricate an alloy consisting of Ag (47%), Sn (30%), Cu (22%), and Zn (1%). An electrical furnace is employed to melt an amalgam alloy inside a controlled inert environment of argon gas to produce alloy specimens, which are further heat-treated at 400 °C for four hours to homogenize the components and phases of the ingot. Subsequently, a prepared cast alloy is pulverized through turning and ball milling, followed by heat treatment at 100 °C for three hours in a vacuum atmosphere for stress reduction. A

chemical study of the base alloy composition is presented in Table 1.

Table 1. the chemical composition analysis for a base alloy and ardent

The Alloy	Ag wt. %	Sn wt. %	Cu wt. %	Zn wt. %
base alloy	47	30	22	1
Ardent	44.5	30	25.5	-----

2.2. Preparation of specimens

Specimens were prepared according to standard No. 1 of ADA [16] by triturating equal quantities of base alloy powder and mercury for 25 seconds using a mechanical amalgamator of the ling-chen type. The specimens exhibit a 1:2 ratio, measuring 4 mm in diameter and 8 mm in height. The specimens were maintained at 37 ± 1 °C.

2.3. Characterization of microstructures - X-Ray diffraction

A base alloy, master amalgam, and their amalgam, all within 2% of Al_2O_3 , have been studied individually using X-ray diffraction techniques to identify the existing phases in the amalgam alloy. The X-Ray machine employed was the SHIMADZU Lab Xe XRD from Japan, with a copper K α radiation generator ($\lambda = 1.5406$ Å) and a nickel filter. The range of the diffraction angle $2\theta^\circ$ extends from 20° to 80°.

Microstructure observation

Specimens were extracted from each sample (base alloy before and after homogenization heat treatment, as well as the amalgam with all additions), ground using various grades of emery paper (400, 600, 800, 1200, 1500, 2000, 2500, 3000), and subsequently polished with 3 μm diamond paste. Specimens were properly cleansed with distilled water and dried using an air blast prior to etching. Polished fill specimens have been etched using a chemical etchant consisting of 30 vol. % nitric acid [17]. Subsequently, it was analyzed under 400x magnification with optical microscopy and scanning electron microscopy (SEM). The distribution of elements on each surface of the fill has been examined both statistically and qualitatively.

2.4. Compressive strength

The Instron machine, namely the UNIVERSAL TESTING MACHINE/WDW 200, has achieved a compressive strength at a rate of 0.5 mm/min [16]. A specimen has been vertically positioned between the jaws. Specimens were tested on the first day and again seven days following the completion of trituration. It must be maintained at 37 ± 1 °C until

testing is completed. The mean value for the five specimens of each amalgam has been documented. The compressive strength can be determined using the following equation [18].

$$\text{Compressive strength} = \frac{\text{Max.force}}{\text{cross sectional area}} \quad (1)$$

2.5. Creep

Specimens prepared for the creep test have been stored, and testing is underway at 37 ± 1 °C. The specimen's length must be measured two hours and forty-five minutes after the conclusion of trituration. The specimen must be exposed to a steady axial pressure of 10 MN/m^2 three hours after the completion of trituration. Furthermore, this load has been sustained for 21 hours. A specimen length was measured using a micrometer caliper, and a reduction was determined by subtracting a second length from the first, which was employed to calculate a percentage of creep. The ADA - Specification No.1 permits a maximum creep percentage of 3%. The creep value for the three specimens has been documented as an average. The creep percentage can be determined using the following equation [16].

$$\text{Creep\%} = \frac{L^0 - L}{L^0} \times 100 \quad (2)$$

Where:

L^0 = original length (mm).

L = final length (mm).

2.6. Vickers micro hardness

The Vickers microhardness of specimens was determined using the digital micro hardness tester (model TH715, Beijing Technology of Time High Ltd) at a static load of 200 g for 10 seconds, conducted at two different time intervals (1 day and 7 days) post-trituration. The upper surfaces of each specimen were abraded using water-lubricated emery paper up to 2000 grit and subsequently polished with diamond paste ($0.25 \mu\text{m}$ particle size) to achieve a clean, consistent finish. Polished specimens were subsequently stored in an incubator at 37 ± 1 °C until testing. At least three indentations have been artificially created at the diametrical distribution of a specimen. The dimensions of an indentation were measured using a microscope at a magnification of $200\times$ when the applied load was removed.

2.7. The thermal conductivity

This particular test has been devised using a thermal setup that featured two copper disks. Specimens were evaluated seven days post-completion of trituration. The specimens must be maintained at a temperature of 37 ± 1 °C until testing is conducted. The specimen is situated

between two top heating disks made of copper. The necessary temperature has been established at 100 °C to compute the coefficient of thermal conductivity. A further reading is acquired, indicating the voltage of the upper and lower copper disks in both cooling and heating scenarios that have been documented. The mean value of four specimens of each amalgam is documented. The heat transfer via conduction is computed using the following equation:

$$q = -k \frac{dT}{dx} \quad (3)$$

The coefficient of thermal conductivity detected depending on an equation as below[9].

$$\lambda = \frac{m c h B}{\pi (R_B)^2 (v_1 - v_2)} (2hp + Rp) / (2Rp + 2hp) \left(\frac{\Delta v}{\Delta t} \right) \quad (4)$$

λ : thermal conductivity coefficient (w/m. k).

m : mass of copper plate (0.824 kg).

C : specific heat capacity of copper plate is 3.805×10^2 (kj / kg .k).

h_p , R_p : thickness and radius of copper plate are 7.01mm, 65mm respectively.

h_B : thickness of specimen (8 mm).

R_B : radius of specimen (2mm).

v_1, v_2 : voltage of thermocouple 1,2 at heating.

Δv : difference in voltage at cooling.

Δt : difference in time at cooling.

2.8. Standard deviation

The standard deviation of a sample set quantifies the dispersion of results relative to the mean. The calculation can be performed by the subsequent formula[19].

$$S^2 = \frac{1}{n-1} \sum_{j=1}^n (x_j - \bar{x})^2 = \frac{1}{n-1} [(x_1 - \bar{x})^2 + \dots + (x_n - \bar{x})^2] \quad (5)$$

Where:

S^2 = standard deviation.

X = individual value.

$\bar{x}$ = arithmetic mean.

N = number of the values.

3. RESULT AND DISCUSSION

3.1. Microstructure characterization

The base alloy utilized for dental amalgam was examined to identify the alloy phases, as illustrated in Figure 1. The phases of the basic alloy were identified by referencing XRD standard cards. The assessment of a phase has revealed that the basic alloy has three phases: γ (Ag_3Sn), ϵ (Cu_3Sn), and β (Ag_4Sn). The γ (Ag_3Sn) represents the matrix of the

microstructure with high pronounced intensity peaks, whereas (Cu_3Sn) shows secondary intensity and denotes a coherent network embedded in the

matrix. The (Ag_4Sn) phase exhibits a relatively low intensity peak in contrast to the peaks of other phases, indicating its scarce presence.

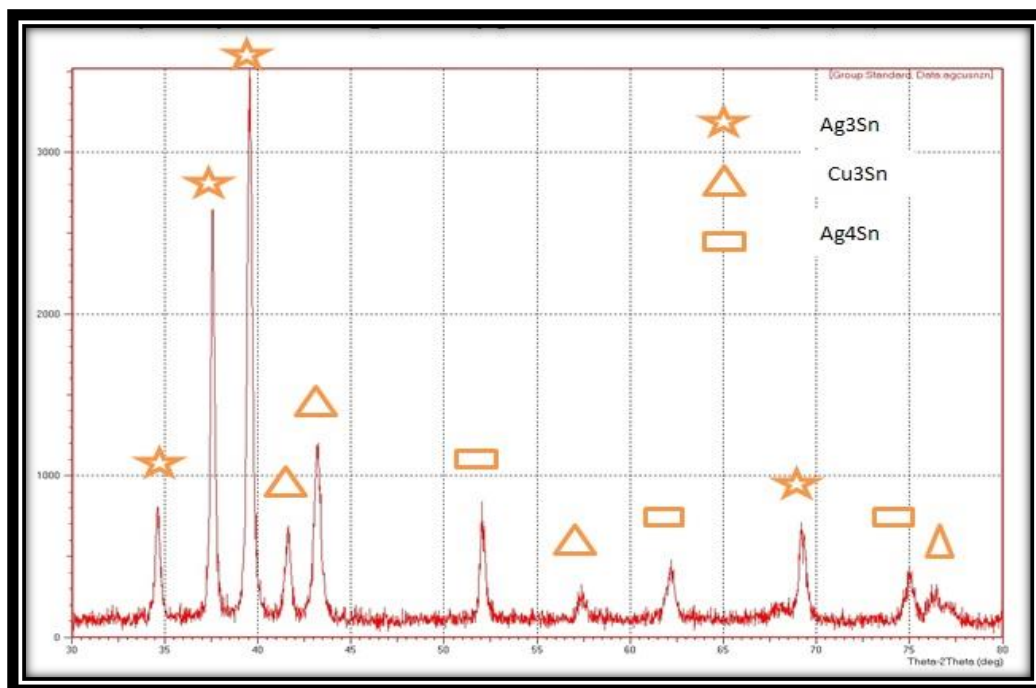
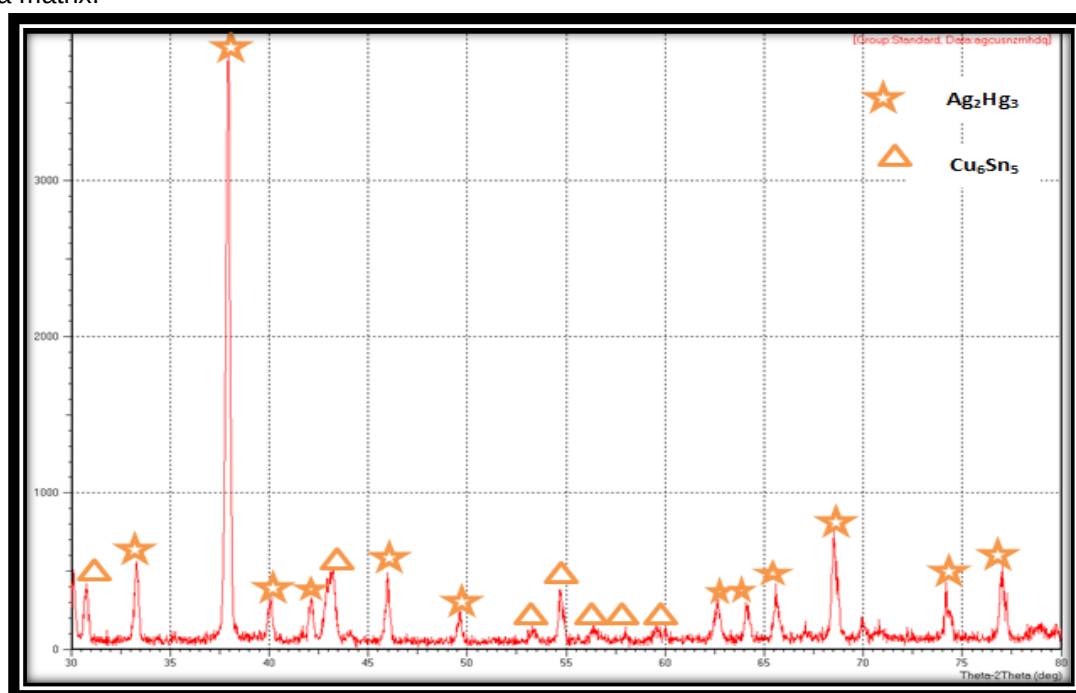


Figure 1. XRD patterns of base alloy

The XRD results of the master amalgam are depicted in Figure 2. The phases of the amalgam have been identified through comparison with conventional XRD cards. An examination of a phase of these amalgams reveals two phases: γ_1 (Ag_2Hg_3) and η (Cu_6Sn_5). The peaks of the γ_1 phase intensity have exhibited maximum intensity high, denotes a matrix of the microstructure, wherein a phase of η exhibits a secondary peak in intensity. This leads to the conclusion that the phase of η signifies the coherent network within a matrix.



Figuer 2. XRD patterns of master amalgam

The XRD results of the amalgam containing 2% aluminum oxide are illustrated in Figure 3. The analysis of a phase for this amalgam reveals three phases: γ_1 (Ag_2Hg_3), unreacted (Al_2O_3), and η (Cu_6Sn_5). The intensity maxima of the γ_1 phase in great quantities exhibit a notable intensity indicative of the microstructural matrix, while the Al_2O_3 phase displays a secondary intensity peak. This will lead to a conclusion wherein the phase of (Al_2O_3) signifies a chaotic network embedded within a matrix.

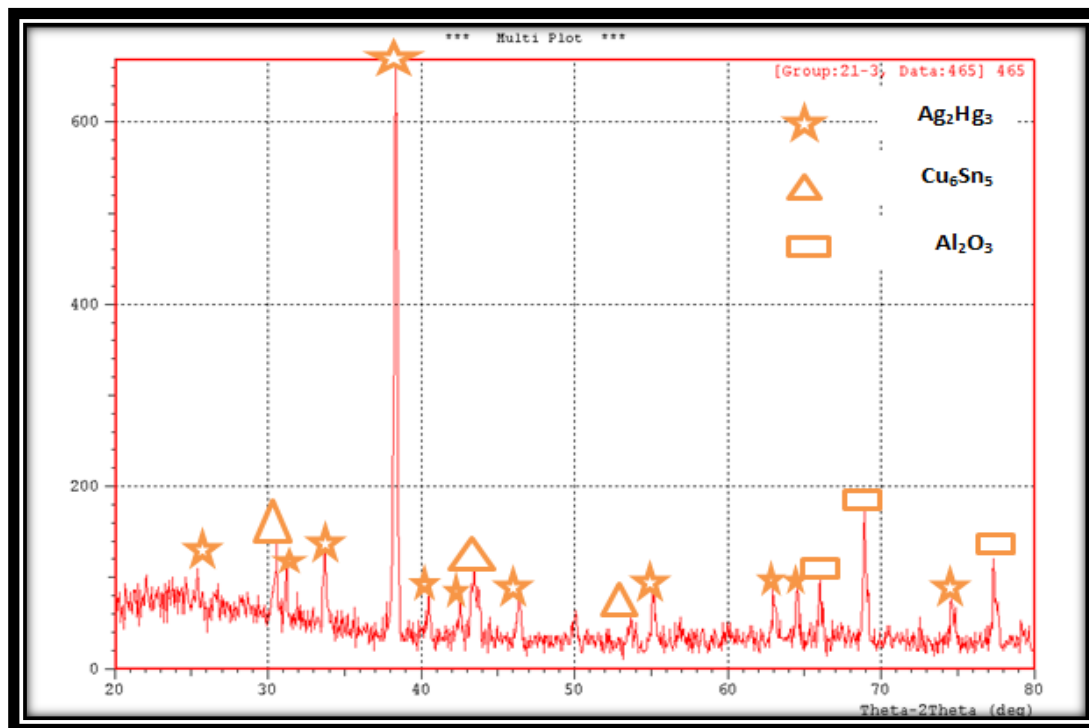


Figure 3. XRD patterns of amalgam with 2% from aluminum oxide

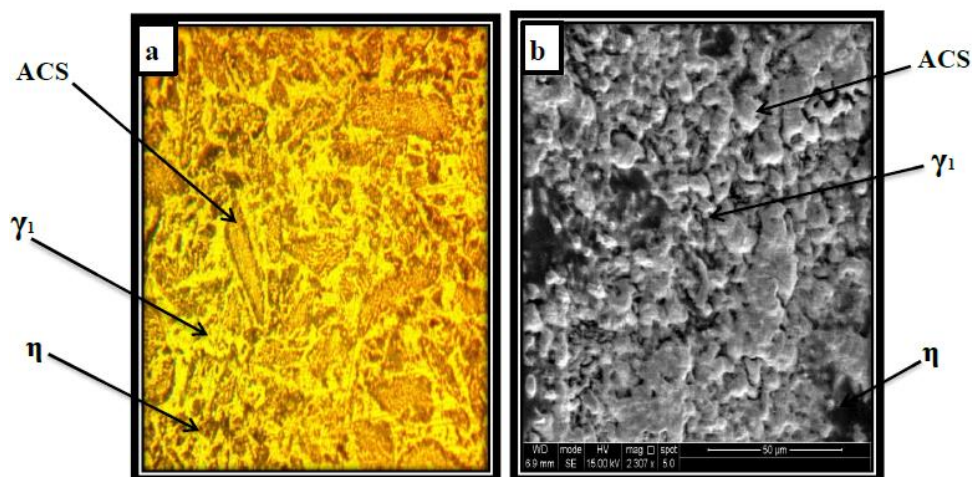


Figure 4. Microstructure observation of the amalgam alloy (M)

a) Via the Optical Microscope at the magnification of (400X), b) Via the SEM where the ACS (Ag-Cu-Sn)

Figures 4–8 illustrate the microstructural examination of the amalgam (M) and its various additions, featuring white areas representing the amalgam matrix of the γ_1 (Ag_2Hg_3) phase and Al_2O_3 phase, alongside black regions indicative of the secondary η (Cu_6Sn_5) phase, and dark gray

regions comprising unreacted ACS (Ag-Cu-Sn) particles. The phases are validated by the X-ray diffraction measurements illustrated in Figures 2 and 3. The aforementioned figures indicate that unreacted particles are encircled by the γ_1 and η phases. Distinct sections can be observed using a

scanning electron microscope (SEM), with the initial region characterized by gray particles. This region comprises elements of silver (Ag), copper (Cu), and tin (Sn) as identified by energy dispersive spectroscopy (EDS). These areas in their results contain unreacted particles. A second portion is depicted in light gray, representing a matrix. In this region, the predominant elements are mercury (Hg)

and silver (Ag) in the γ_1 (Ag_2Hg_3) phase high quantities, along with the Al_2O_3 phase. In the same region, the Cu level is elevated, and Sn has been identified, representing the η phase. The size of unreacted particles (ACS) grows with the rising percentages of (Al_2O_3) due to the presence of (Al_2O_3) powder. This powder reduces the diffusion of mercury to the particles of the alloy powder.

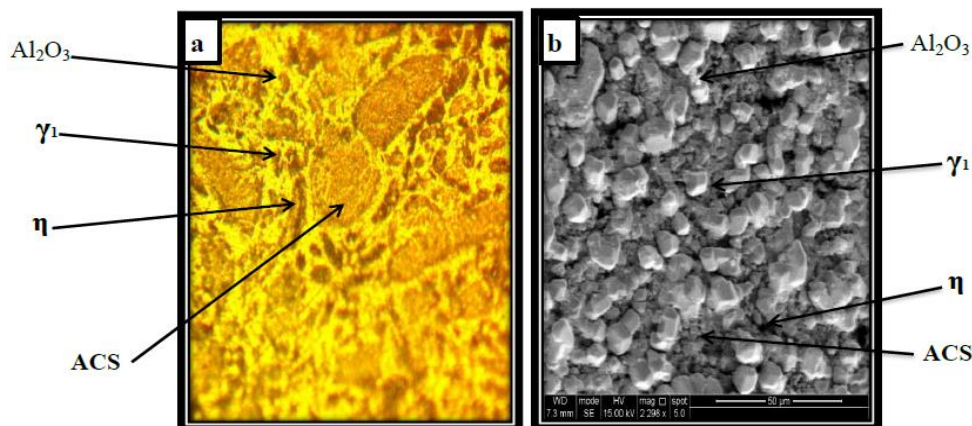


Figure 5. Microstructure observation of the (0.5% Al_2O_3) amalgam
a) Via the Optical Microscope at the magnification of (400X), b) Via the SEM where ACS (Ag-Cu-Sn)

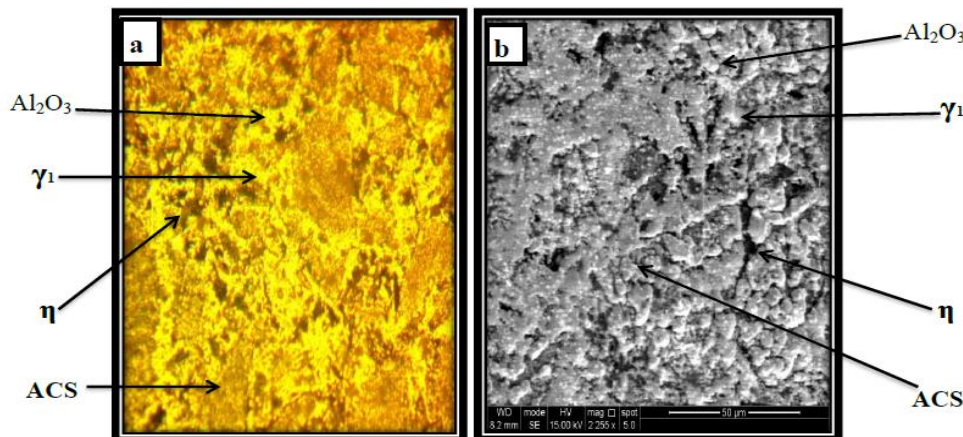


Figure 6. Microstructure observation of the (1 % Al_2O_3) amalgam
a) Via the Optical Microscope at the magnification of (400X), b) Via the SEM where the ACS (Ag-Cu-Sn)

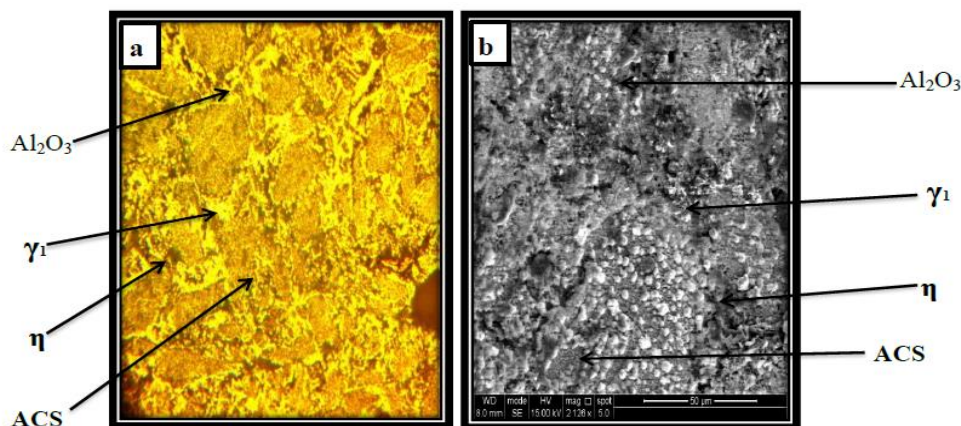


Figure 7. Microstructure observation of (1.5% Al_2O_3) amalgam
a) Via the Optical Microscope at the magnification of (400X), b) Via the SEM where the ACS (Ag-Cu-Sn).

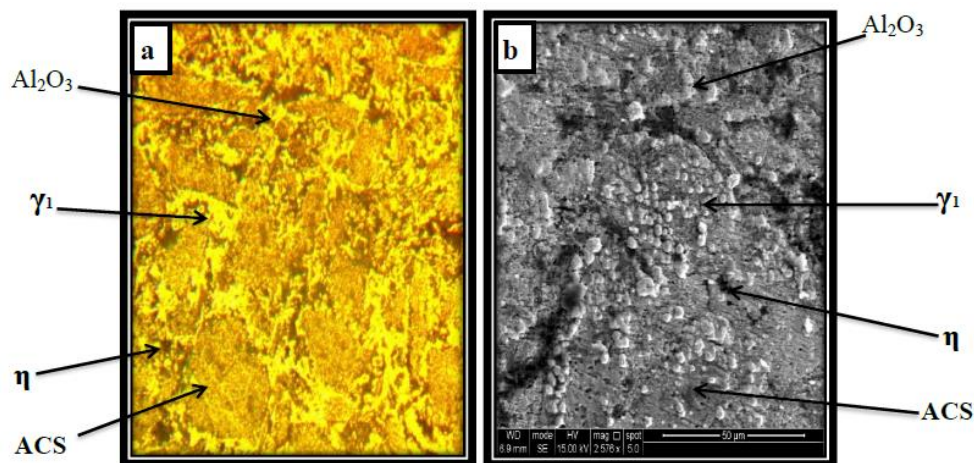


Figure 8. Microstructure observation of (2 % Al_2O_3) amalgam

a) Via the Optical Microscope at the magnification of (400X), b) Via the SEM where the ACS (Ag-Cu-Sn)

3.2. The compressive strength

The mean value of compressive strength results and the standard deviation have been reported by using five specimens mean of each (ardent, M amalgam and amalgam with additions) into two different aging times (1 day and 7 days) after the trituration is completed are presents in the Table 2.

Table 2. The mean value, standard deviation of compressive strength at two different aging time and improvement percentage of the (7 days)

Alloys	Mean Compressive Strength (MPa)				Improvement Percentage%
	1 day	S.D.	7 days	S.D.	
Ardent	36.5	1.5	41.5	1.5	-----
M	39	1	45	1	-----
0.5%	41.5	2.5	48.5	0.5	17
1%	46.5	1.5	52	2	26
1.5%	51	1	57.5	1.5	39
2%	54.5	0.5	65	1	57

The enhancement in compressive strength at 7 days compared to 1 day is observed, since the setting reaction of amalgams remains incomplete. The incorporation of (Al_2O_3) enhances the compressive strength as this inclusion generates a metal matrix composite (MMC) where the particulate phase exhibits greater hardness and rigidity than the matrix. These reinforcing particles generally inhibit the mobility of the matrix phase surrounding each particle. The highest enhancement percentage recorded has been 57% with a 2% (Al_2O_3) incorporation.

The reduced compressive strength relative to the minimum standards of ISO 24234 for high-copper dental amalgam may be attributed to internal porosity and microstructural flaws developed during specimen fabrication. These imperfections can act as sites of stress concentration, promoting the onset and advancement of cracks under compressive forces, hence resulting in a decreased compressive strength.

3.3. Creep test

Results that obtained from the mean value of creep and standard deviations of the three specimens for each (ardent, M amalgam and amalgam with additions) are presented in Table 3. acceptable value of the creep in the dental amalgam according the ADA specification No.1 must be no more than 3% [17].

Table 3. the mean value, standard deviation and improvement percentage of the creep test

Alloys	Mean Creep (%)	S.D	Improvement Percentage%
Ardent	0.94	0.045	-----
M	0.875	0.045	-----
0.5%	0.695	0.065	21
1%	0.49	0.01	44
1.5%	0.38	0.08	57
2%	0.23	0.02	74

The effect of (Al_2O_3) addition on creep resistance of the amalgam has followed up the same direction as on the compressive strength. the percent for a creep of amalgam decreased through the increasing in the content of the (Al_2O_3) until it reaching a minimal (the lowest) value

(0.23%) for 2% (Al_2O_3) and improvement percentage reach 74%. The presence of solid alumina (Al_2O_3) particles impedes the movement of grains and the matrix's deformation under sustained loads, hence reducing the material's deformability.

3.4. Micro hardness test

The micro hardness test results and the standard deviation have been obtained by four specimens of each (ardent, M amalgam and amalgam with additions) in two different aging time (one day and seven days) from the end of trituration are shown in Table 4.

Table 4. The mean value, standard deviation of the hardness test at two different aging time and improvement percentage of the 7 days

Alloys	Vickers Micro hardness (HV)				Improvement Percentage %
	1 day	S.D	7 days	S.D.	
Ardent	122.2	1.8	146.1	1.85	-----
M	126.5	1.1	143.5	1.6	-----
0.5%	123.7	2.3	167.9	0.45	17
1%	139.8	1.4	172.3	1.1	20
1.5%	147.3	0.9	181.8	3.4	27
2%	166.7	1.5	192.4	4.1	34

It's obvious from the table above, like all other mechanical properties and in the same trend, that there is a significant increase in the hardness after 7 days aging as compared to 1 day test this is because the setting reaction doesn't complete and may be some unreacted mercury present. Mercury will weaken the amalgam. Thus after one week amalgam reaches its final strength, where mercury is Completely depleted, and forms the final phases. The addition of (Al_2O_3) increases the hardness of amalgam 2% (1 day 166.7 HV, 7days 192.4 HV) and improvement percentage reach 34% as compared with the master (1 day 122.2 HV, 7days 146.1 HV). This phenomenon arises from the reinforcing influence of Al_2O_3 on the amalgam; alumina is a ceramic substance characterized by significant hardness that obstructs dislocation movement.

3.5. Thermal conductivity

The thermal conductivity test results and the standard deviation were obtained by four specimens of each (ardent, M amalgam and amalgam with additions) in 7 days of aging time after the trituration is completed are shown in Table 5.

Table 5. The value of mean, standard deviation and improvement percentage of the thermal conductivity test

Alloys	Mean Thermal Conductivity (W/m. k)	S.D.	Improvement Percentage%
7 days			
Ardent	77.85	0.35	-----
M	76.9	1.5	-----
0.5%	60	1	22
1%	55.05	0.15	29
1.5%	42.75	2.75	45
2%	33.15	0.75	57

The data in Table 5 indicates that the master exhibits the highest value in comparison to other amalgams, as an increase in Al_2O_3 content leads to a decrease in thermal conductivity due to Al_2O_3 's inherently low thermal conductivity. The thermal conductivity of Al_2O_3 ranges from 28 to 35 W/m-K; so, including Al_2O_3 powder into the amalgam would diminish thermal conductivity, and as the proportion of Al_2O_3 increases, thermal conductivity further declines. Thus, this will mitigate the heat shock to the dental pulp. The highest percentage of enhancement in the decrease of thermal conductivity is 57% at 2% (Al_2O_3).

4. CONCLUSION

1 - Alumina addition enhanced a compressive strength of the amalgam, which increases with increasing Al_2O_3 content, where the maximum strength value was (65MPa) at 2% (Al_2O_3) with improvement percentage of 57%.

2. Alumina addition improved the creep resistance of the amalgam which increases with increasing Al_2O_3 content, where the minimum creep value was (0.23%) at 2% (Al_2O_3) with improvement percentage of 74%.

3 Alumina addition improved the micro hardness (HV) of the amalgam, which increases with increasing Al_2O_3 content, where the maximum hardness value was (192.4 HV) at 2% (Al_2O_3) with improvement percentage of 34%.

4 Alumina addition enhanced The thermal conductivity of the amalgam, which decreases with increasing Al_2O_3 content, where the minimum thermal conductivity value was (33.15 W/m. k) at 2% (Al_2O_3) with improvement percentage of 57%.

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IZVOD

UTICAJ DODAVANJA MIKRO- Al_2O_3 NA ČVRSTOĆU NA PRITISAK, PUZANJE, MIKROTVRDOĆU PO VIKERSU I TOPLOTNU PROVODLJIVOST ZUBNOG AMALGAMA SA VISOKIM SADRŽAJEM BAKRA

Glavni cilj ovog istraživanja je proučavanje uticaja dodavanja (Al_2O_3) na (čvrstoću na pritisak, mikrotvrdoću po Vickersu, puzanje i toplotnu provodljivost) zubnog amalgama sa visokim sadržajem bakra. Livenje je korišćeno za stvaranje jedne legure sastavljene od Ag (47%), Sn (30%), Cu (22%) i Zn (1%). Dodatak (Al_2O_3) se vrši u različitim procentima (0,5, 1, 1,5, 2 tež. %). Prilikom pripreme uzoraka praćena je ADA specifikacija br. 1. Uzorci su čuvani na $37 \pm 1^\circ \text{C}$ u staklenoj komori posebno dizajniranoj za ovu svrhu. Pritisna čvrstoća amalgama se povećava sa dodatkom od 0,5% do 2% (Al_2O_3), puzanje amalgama se smanjuje nakon dodatka od 0,5% do 2% (Al_2O_3), a mikrotvrdoća HV amalgama se povećava sa povećanjem sadržaja Al_2O_3 . Termička provodljivost amalgama se smanjuje sa dodatkom od 0,5% do 2% (Al_2O_3).

Ključne reči: Zubni amalgam. pritisna čvrstoća, puzanje, Vickersova mikrotvrdoća; Al_2O_3 , toplotna provodljivost

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