# Improvement of Dental Amalgam Properties By Increasing Copper Content

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**ABSTRACT**-the aim of the present investigation is to study the effect of copper content on mechanical properties of dental amalgam. For this purpose five alloys have been casted composed of constant percentage of tin 30wt% and the copper content is vary as follow(5, 10, 15, 20, 25) and the rest was silver. The specimens were prepared and tested according to ADA specification No. 1. Mechanical properties (compression strength, diametral tensile strength, creep, dimensional change, and hardness) were investigated and it is found that all mechanical properties enhanced by increasing copper content. The compression strength is increased by 37.4% at one day and 35.2% at 7 days test. The diametral tensile strength is increased by 18.1% at one day and 13.5% at 7 days test. Creep resistance is increased by 71.1%. Vickers microhardness increases by 29.4% at one day test and 27.3% at 7 days test. Increasing copper content stabilizes the dimensional change of dental amalgam. All as compared to low copper content amalgam.

## 1 INTRODUCTION

Dental amalgam, in widespread use for over 150 years, is one of the oldest materials used in oral health care[1]. It has been estimated that 75 % of all single tooth restoration are amalgam restoration and that this percentage has remained stable for many years[2]. For an amalgam restoration to be successful, it must be stable and resistant to the solvents and stresses of the oral environment. Several characteristics of the material are important such as susceptibility to corrosion, dimensional change, creep, strength, elastic modulus, and fracture resistance, determine the structural stability of the material[3].

Compressive and tensile strengths of core materials are thought to be important because core build-ups usually replace a large amount of tooth structure and must resist multidirectional masticatory forces for many years[4]. The value of strength of restorative materials should be close to the value of strength of the tooth structure. Compressive strength is only one of the criteria for the selection of core material, but it is a crucial one[5].

Creep is the time-dependent inelastic deformation of materials that are used at temperatures that are close to their melting points. Expressed in absolute temperatures, the melting point of the major matrix phase (γ1) in dental amalgam is 400 K, whereas it is used at the mouth temperature of 310 K for a ratio of 0.8. In metals, ratios that exceed 0.5 are considered to be a forerunner for examining creep behavior[6].

The setting reaction for amalgam involves a dimensional change. The overall effect may cause a slight final expansion or a slight final contraction [7]. Excessive contraction lead to a large marginal gap between the material and the cavity[8]. Expansion lead to extrusion of the material out of the cavity[9].

These characteristics are influenced by several factors, including Hg/alloy ratio, condensation technique (i.e., the amount of porosity), and trituration conditions[10,11].

Hasheminezhad et al. [12] investigated the effect of copper content on compressive strength of dental amalgams. Addition of copper content to dental amalgam alloy causes an increase in compressive strength and hardness. Gaurav Solanki [13]found that high copper containing amalgam was be much better than low copper alloy containing amalgam in respect to strength, corrosion resistance, durability and resistance to tarnish. The tooth restored with high copper amalgam did not have any kind of marked expansion or condensation after its setting after 24 hrs. creep were also minimized.

The aim of this work is to investigate the effect of copper content on mechanical properties of dental amalgam.

## 2. EXPERIMENTAL

### 2.1 Powder Alloy Preparation

The element of amalgam alloys (silver, copper, tin and zinc) are melted using electrical furnace and the operation was done under inert atmosphere using argon gas. Five alloys have been casted composed of constant percentage of tin 30% and the copper content is vary from 5 to 25% with interval of 5%. Copper is increased in the expense of silver. The ingots that have been obtained by casting were heat treated at 400Co for 4 hour to obtain uniform distribution of the ingot elements and phases. The prepared cast alloys are subsequently transformed into powder. This transformation has been done by ball mill of ceramic balls and polyethylene jar to obtain a mean particle size of 40μm. Finally the powder heat treated for stress relief at 100oC for three hour under vacuum atmosphere. The chemical composition of the alloys is expressed in Table 1.

Table 1 Illustrates the chemical composition analysis of the used alloys.

| Alloy | Ag wt% | Cu wt% | Sn wt% | Zn wt% |
|---|---|---|---|---|
| A | 66.79 | 4.95 | 24.84 | 2 |
| B | 61.1 | 9.83 | 27.4 | 1.82 |
| C | 56.2 | 15.3 | 23.72 | 2.4 |
| D | 53.69 | 19.3 | 25.63 | 1.8 |
| E | 47.81 | 23.41 | 26.6 | 1.6 |

### 2.2 Specimens Preparation

The specimens were made according to the ADA specification No.1 [14] by trituration of equal amount of alloy powder and mercury for 30 sec by mechanical amalgamator type (ling chen). The dimensions for the specimens is 1:2 (4 mm in diameter and 8 mm in height). The specimens have been stored at 37±1 $^{o}$C.

### 2.3.Microstructure

A specimen of each amalgam were taken and prepared for microscopic examination using the standard metallographic procedure. Rough polishing was done on successively finer grades of emery papers (360,400, 600, 800, 1000, 1200, 1500, 2000),then polished with diamond past of 0.25 μm particle size. The specimens were then washed thoroughly with distilled water and dried in an air blast.

Amalgam specimens were etched using the following solutions shown in Table 2[15]:

TABLE 2

PRESENT THE ETCHING SOLUTIONS.

| Solution A | Solution B | "Hypo" rinse |
|---|---|---|
| 4 gm. $K_2Cr_2O_7$ | 4 gm. I | $Na_2S_2O_3$ .5$H_2O$ |
| 1 gm. KI | 96 ml. ethyl | Dissolved in water |
| water100 ml. $H_2O$ | alcohol | |



The sample was swabbed with Solution A for 20-40 seconds and rinsed with water. Then it was lightly swabbed with Solution B for 5-15 seconds, followed with a "hypo" swab-rinse, washed with water, and dried.

Inverted metallurgical microscope is used at 400x magnification in the microstructure examination of the amalgam specimens.

### 2.4 Mechanical Properties

Compressive strength and diametral tensile strength tests were carried out with a universal testing machine type WDW 200, China. The tests were run at a constant loading speed of 0.5mm/min. The first measurement was done 1 day after the end of trituration; the second was measured after 7 days. Meanwhile these specimens where stored at a constant temperature of 37±1 oC until the tests were done. For compressive strength the specimen was placed vertically between the jaws and its calculated by using the following equation :-

$$\text{Compressive strength(MPa)} = \frac{Max.force(N)}{cross\,sectional.area(mm\text{^}2)}$$

For diametral tensile strength the specimen was placed on the lateral side rather vertically and its calculated by using the following equation:-

$$\sigma_t = \frac{2P}{\pi DL}$$

where

P = load at fracture (N).

D = diameter of specimen (mm).

L = length of the specimen (mm).

σt = tensile strength MPa

In order to measure the creep, the prepared specimens were stored in an incubator maintained at 37± 1oC. Before running the test, the area of the two sides of the specimen was grinded with emery paper. Two hours and 45 minutes after the end of trituration the length of the specimen should be measured. At three hours after the end of the trituration the specimen should be subjected to a constant axial pressure of 10MN/m2 were applied at constant temperature of 37± 1 °C. This was done in a continuous manner for 21 hours after which the specimen length was measured again.

For dimensional change the first record was taken immediately after specimen was formed (30 minutes after trituration), the specimen was placed in a measuring instrument (micrometer) having an accuracy 0.1µm. The specimens were sit free without any restraint during the test. The length of the specimens was measured after 24h after the end of trituration. The specimens were kept at a constant temperature of 37± 1oC at the incubator. According to the A.D.A. specification No.1 the dimensional change must be within range of ± 20 µm/cm.

Vickers microhardness tested in a digital Microhardness tester (Type TH715, Beijing, Time High Technology Ltd), at a static load of 200 g for 20 seconds, that was performed in different time intervals (24 hour and 7 days) after the end of trituration. The top surfaces of each specimen were ground with water-lubricated emery paper up to 2000 grit paper and polished with diamond past(0.25 µm particle size) to produce a smooth, uniform surface. Polished specimens were then stored in incubator at 37± 1 °C till testing. At least three indentations were made at diagonal distribution across the specimen.

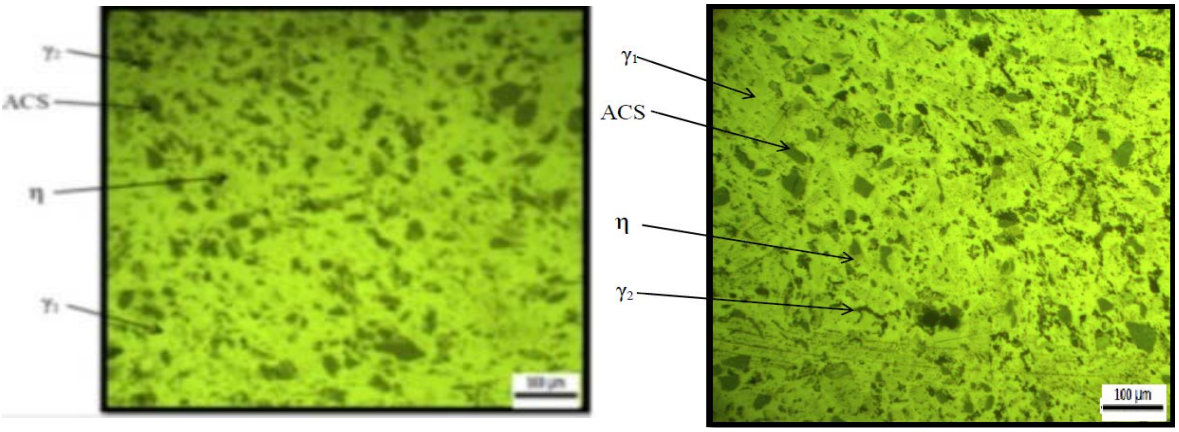
## 3. RESULT AND DISCUSSION

The microstructure of amalgam illustrated in Figure 1 represent the results obtained with the iodine etch in which the phases can be distinguished at one time. The unreacted particles ACS (Ag-Cu-Sn) appear gray which surrounded with η phase and a continuous γ1 matrix and embeds γ2 phase if any.

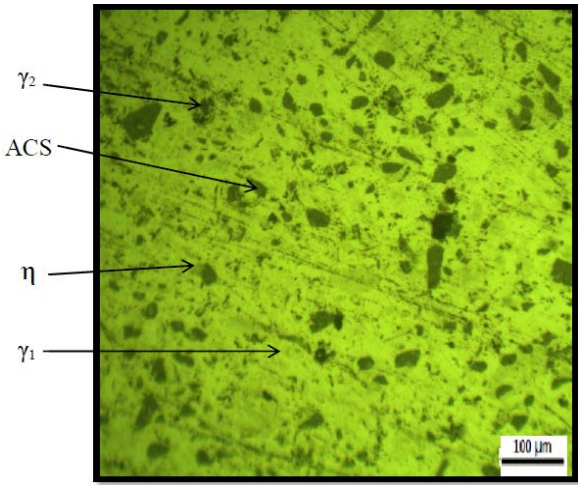
The γ1 phase appears as a matrix of light gray color. The γ2 areas etch in dark and are distinguishable from the voids. When viewed in the microscope, the voids show as black, out of focus areas. Because the amount of liquid mercury used to amalgamate with the alloy particles is less than that required to complete the reaction. Thus, the final microstructure of the set amalgam mass consists primarily of unreacted particles surrounded by a matrix of the reaction products. The unreacted particles is bonded together by the matrix.

Comparison of microstructure of all amalgams relative to each other shows γ2 phase amount decreases to become vanished and η phase amount increases as copper content increases.

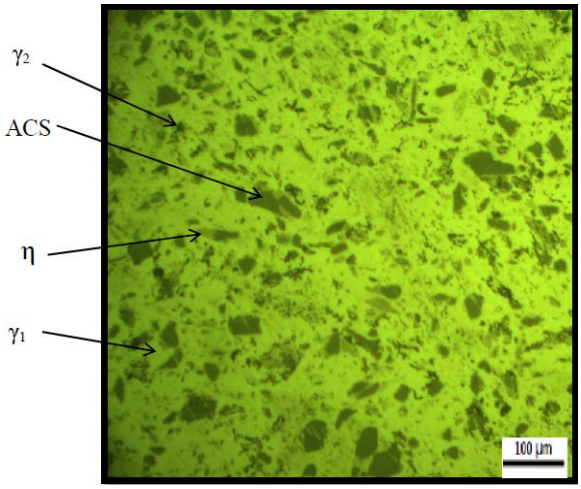




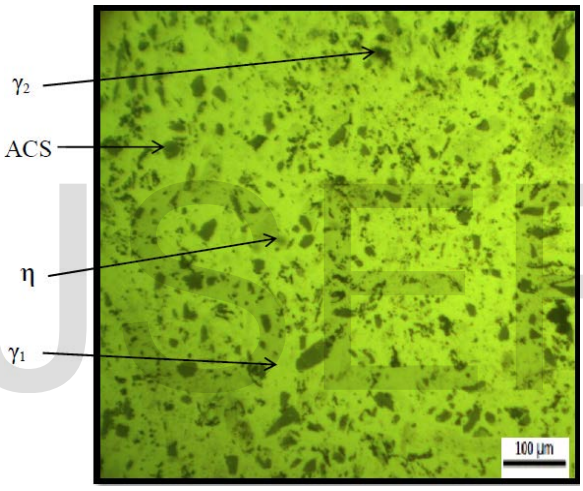

Amalgam (A) Amalgam (B)




Amalgam (C)




Amalgam (D)




Amalgam (E)

**Figure 1 The microstructure of amalgams.**

The mean value of compression strength and diametral tensile strength results were reported by using four specimens mean of each amalgam in two different aging time (1day and 7 days) from the end of the trituration are shown in Figure 2 and 3 respectively. Its noted that there is a significant increase in the compression strength and diametral tensile strength after 7 days aging as compared to 24 h test this is all because that the setting reaction doesn't completed and may be some unreacted mercury present. Mercury will weaken the amalgam. Thus after one week amalgam reach its final strength, where mercury is completely depleted, and forming their final phases.

Also the addition of copper increases the compression strength and diametral tensile strength. This increase in compression strength and diametral tensile strength can be explained in term of phases formed after amalgamation since crack always initiated and propagated in the matrix. This is shown in Figure 4. Even in the matrix the crack will initiated and propagated in the weakest phase. Thus the relative weakness of the lower copper content amalgams from the presence of γ2 phase which is the weakest phase present in all of the amalgams studied.

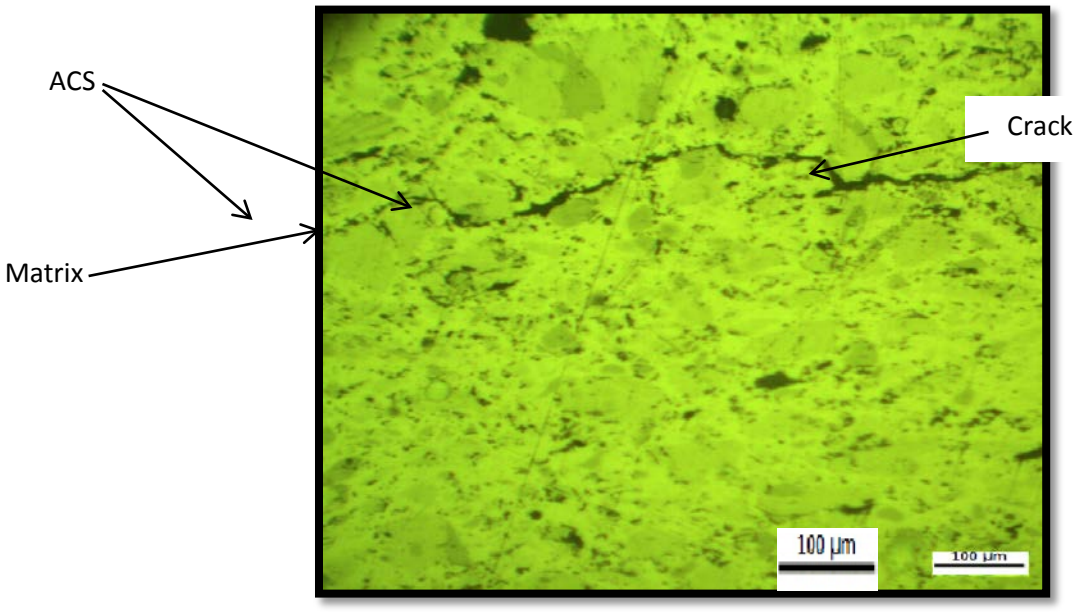



**Figure 4 The regime of the crack E.**

Increasing copper content reduces γ2 phase and a crack has to initiate and propagate in γ1 which is stronger than γ2 phase. So, having more of the stronger phase having stronger amalgam.

The results obtained from the mean creep value of two specimens of each amalgam are shown in Figure 5. According to the ADA specification No. 1, the acceptable creep value in a dental amalgam should be less than 3%. The creep value of all present amalgams were lower than ADA limit (3%). The effect of copper addition on creep resistance of the amalgam followed the same trend as on compressive strength and diametral tensile strength. As indicated in Figure 5, the creep percent of the amalgam decreased with increasing copper content until reaching the lowest value (0.13%) for E amalgam (25wt% copper). The presence of the γ2 phase is associated with higher creep rates. In addition to the absence of the γ2 phase, the very low creep rates in single composition high copper amalgams may be associated with η rods, which act as barriers to deformation of the γ1 phase. For these reasons the resistance to creep is increases with increasing copper content of amalgam. Also, its noted with all amalgam tested that there is a reduction in volume of the tested specimens. This reduction is resulted from the closer of the porosity that is formed during trituration and from the condensation processes.

The mean value of dimensional change were obtained by three specimens of each amalgam are shown in Figure 6. ADA specification No.1 determines the allowable dimensional change is ±20 µm/cm. The result shown in Figure 6 indicated that all the amalgams within the allowable limit set by the ADA and the dimensional change of amalgam stabilized with increasing copper content. The minimum dimensional change is 6.6 µm/cm for E amalgam.

The microhardness test results were obtained by three specimens of each amalgam in two different aging time (1 day and 7 days) from the end of the trituration are shown in Figure 7. It's obvious from the above table, 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 all because that the setting reaction doesn't completed and may be some unreacted mercury present. Mercury will weaken the amalgam. Thus after one week amalgam reach its final strength, where mercury is completely depleted, and forming their final phases.

Also the addition of copper increases the hardness of amalgams as compared with the low copper amalgam. This increase in hardness is due increasing copper content reduces γ2 phase and increases η phase.

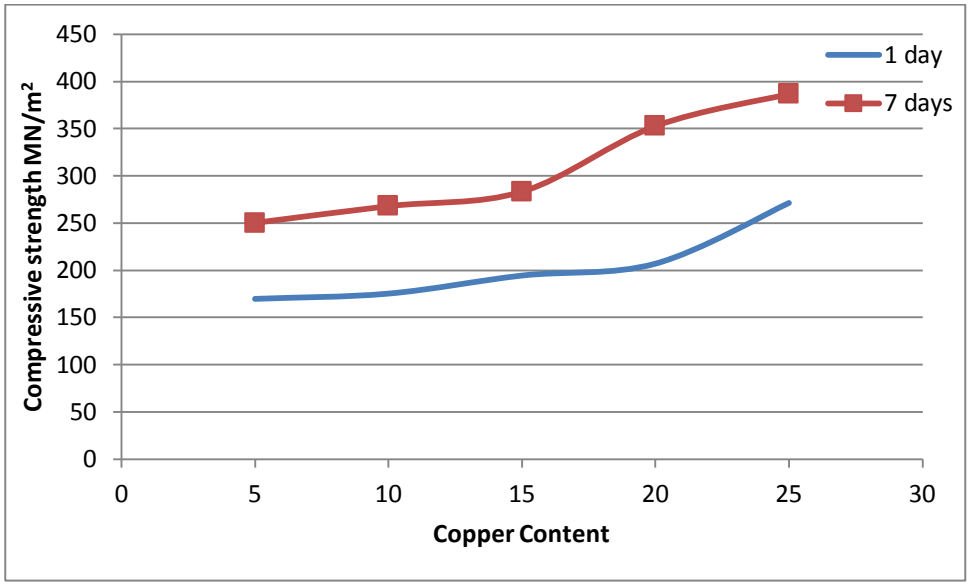



**Figure 2 The mean value of compressive strength at different aging time.**

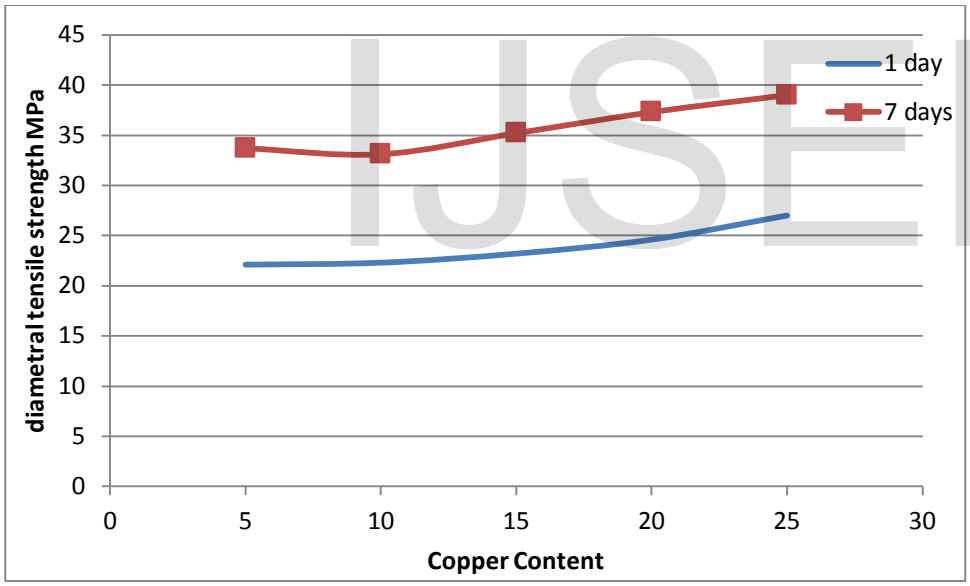





**Figure 3 The mean value of diametral tensile strength at different aging time.**

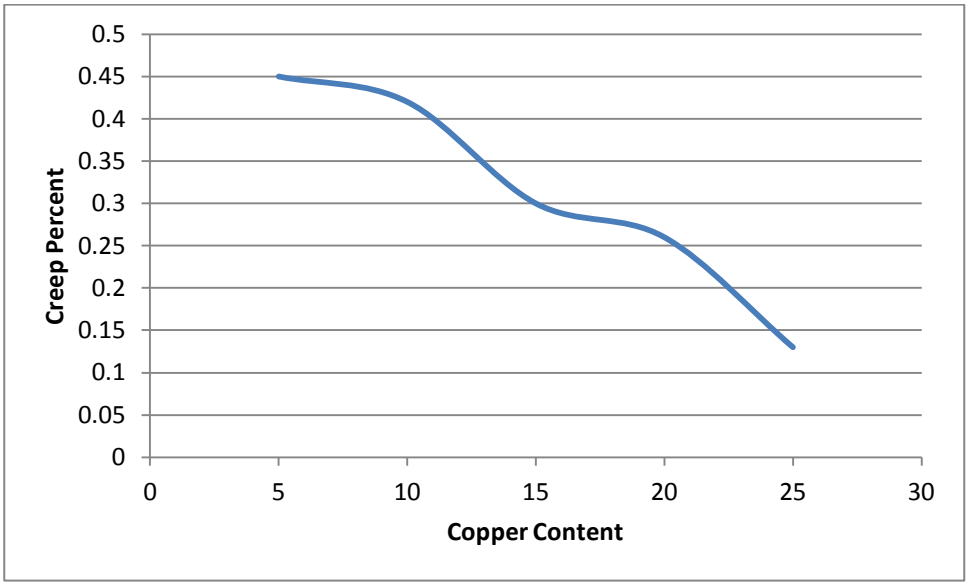



**Figure 5 The mean value of the creep test.**

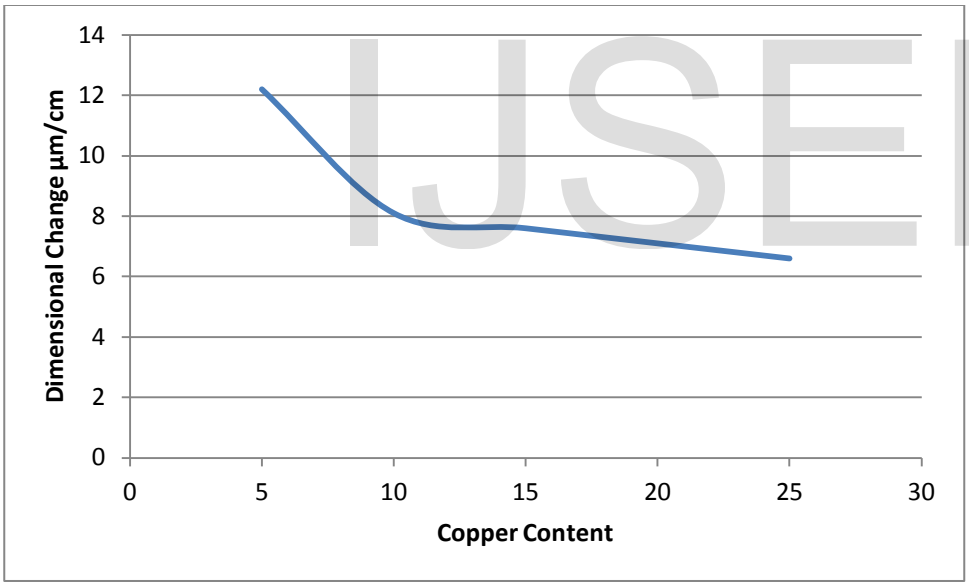





**Figure 6 The mean value of dimensional change test.**

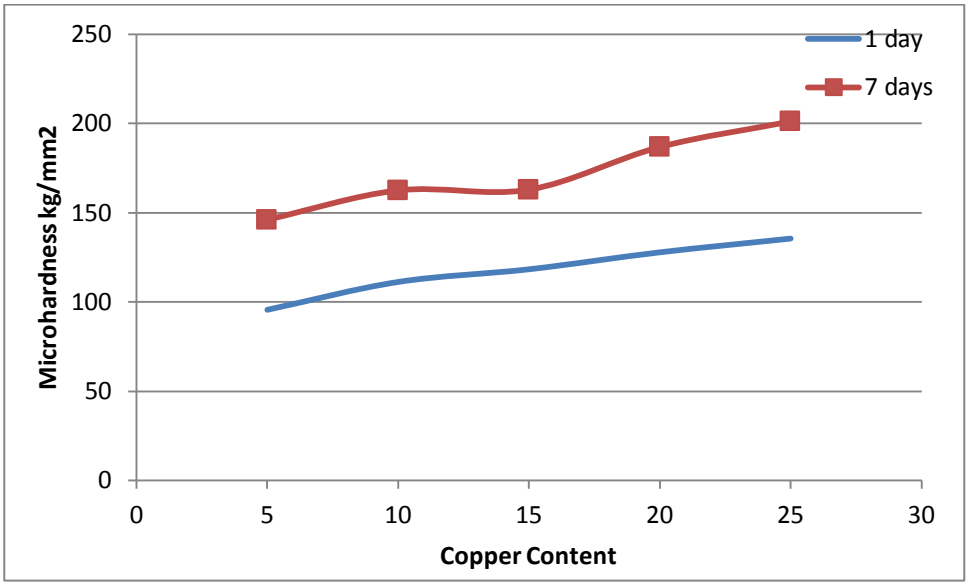



**Figure 7 The mean value of Vikers microhardness test at different aging time.**

## 4. CONCLUSION

1. Increasing copper content enhance the compression strength by 37.4% at one day and 35.2% at 7 days test as compared to low copper content amalgam.
2. Increasing copper content enhance the diametral tensile strength by 18.1% at one day and 13.5% at 7 days test as compared to low copper content amalgam.
3. Increasing copper content increases the creep resistance by 71.1% as compared to low copper content amalgam.
4. The Vickers microhardness increases by 29.4% at one day test and 27.3% at 7 days test as compared to low copper content amalgam.
5. Increasing copper content stabilizes the dimensional change of dental amalgam.



## 5. REFERENCES

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