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Experimental and Numerical Study of Thermal Properties for Amalgam Reinforced by NiCrMo Nanowires

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ABSTRACT

NiCrMo nanowires with four weight percents include 0.3, 0.6, 0.9 and 1.2 wt% have been added to conventional amalgam to improve its properties especially thermal conductivity which is represented important property to reduce the sensation of root for hot and cold foods and beverages. NiCrMo nanowires were selected because they closed in composition to the known dental alloy (Ni-Cr alloy). SEM technique was used to characterize the modified structure of nanocomposites.

The thermal conductivity was decreased after adding NiCrMo nanowires to amalgam and the reduction is increases with increasing wt% of nanowires, this is due to the dominance of smooth temperature dependence by nanowires which suggest a phonon-phonon or phonon-defect scattering.

AFM inspection also was achieved to show the morphology of surface in addition to measure roughness, and the results showed the decreasing in roughness of amalgam/nanowires composites compared with amalgam due to decreasing in surface irregularities by adding nanowires. This reduction in roughness leads to decrease the accumulation of bacteria on amalgam and then reducing biocorrosion. Also, microhardness of amalgam/NWs composites was increased compared with conventional amalgam and the increment increased with increasing wt% of nanowires. The increment in microhardness is suggested the filling of vacancies within amalgam by nanowires and getting more coherent for components in amalgam especially with Hg which is the main cause of the reduction in hardness. On the other word, in present work, we improved thermal conductivity in addition to get more microhardness and less surface roughness by adding nanowires. Simulation study was achieved for thermal properties to confirm the role of NiCrMo nanowires to reduce heat

دراسة نظرية وتجريبية للخواص الحرارية للملغم المقوى باسلاك ثانوية ذات اساس NiCrMo

الخلاصة

تم إضافة أربعة نسب مختلفة من الأسلاك النانوية المصنعة من سبيكة NiCrM 1.2 , 0.9 , 0.6 , 0.3 إلى الحشوات الزئبقية التقليدية لتحسين خواصها وخصوصا خواص التوصيل الحرارية لتقليل تحسس جذر السن للمأكولات او المشروبات الحارة او الباردة. وتم اختيار أسلاك نانوية من سبيكة NiCrMo وذلك لكونها إحدى السبائك الطبية التي يتم استخدامها في تطبيقات الأسنان.

نلاحظ انخفاض التوصيل الحراري للحشوات الزئبقية بعد إضافة الأسلاك النانوية ويزداد الانخفاض بزيادة النسب المضافة وذلك بسبب الاستطارة التي تحدث بين فوتون – فوتون او فوتون – العيوب.

في فحص AFM تم دراسة طبيعة السطح لعينات الحشوات الزئبقية وقد لوحظ انخفاض الخشونة السطحية بزيادة نسبة الأسلاك النانوية المضافة وهذا الانخفاض في الخشونة السطحية مفيد لمنع تجمع البكتريا على سطح الحشوات الزئبقية وبذلك يقلل معدل التآكل البايلوجي , كذلك زادت نسبة الصلادة للحشوات الزئبقية بزيادة نسبة الأسلاك النانوية المضافة وذلك

الكلمات المفتاحية:
اسلاك نانوية,التحليل الحراري NiCrMo, الملغم التقليدي.

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Introduction

The transition of some heat from dental materials to the mucosal surfaces is undesirable, also these materials can have sensation of hot/cold and the filling material should not transmit heat to the pulp of the tooth. Amalgam is the oldest material which used for restoration and it is transfer some heat to other parts in mouth, and then the patient will feel with pain.

Amalgam is a mixture of metals (Ag, Sn and Cu) with liquid Hg, this mixture include some phases such as Ag-Sn alloy which dissolves in the liquid Hg, and forming the new phases that then are solidified. Significant amounts of the unreacted Ag_3Sn (γ phase) also are included in amalgam in addition to Cu_3Sn phase and Hg, the letter is reacts with Ag-Sn alloy to form the desirable γ_1 phase (Ag_2Hg_3). Finally, the amalgam is seemed as paste.

Many authors fabricated Ni nanowires and its alloys because of their novel properties to use them for various applications. These nanowires included Ni-rich/Cu multi-layered nanowire [1], Au/Ni multilayered nanowires [2], Ni-Co and Ni-Co/BaFe nanowires [3], NiSi nanowires [4], intermetallic NiAl nanowire [5], single crystalline Mo nanowires [6], NiFe nanowires [7]. On other hand, Po-Hsien et al. studied some mechanical properties of NiTi nanowires under the nanobending using atomic force microscopy with molecular dynamics simulations [8]. Ni nanowires with length of 11µm and diameters 800 and 15nm also grow within the pores of nuclear track polycarbonate membrane by electrodepositing nickel [9]. Reza et al. studied the molecular dynamics simulations of structural transformations in NiTi nanowires [10]. In addition to fabricate Si nanowires [11], NiMo nanowires [12].

Many researchers highlighted on improving properties of amalgam by adding nanostructured materials such as nano ZnO and Alumina [13] and TiO_2 nanoparticles[14].

Because of the importance of NiCrMo alloy in a dental application, we investigated on using NiCrMo nanowires as reinforcement for amalgam as attempt to improve thermal conductivity in addition to investigate microhardness and roughness of surface after adding four weight percents of NiCrMo nanowires (0.3, 0.6, 0.9 and 1.2wt). The nanocomposites were characterized by SEM to investigate the change in microstructure.

Experimental Procedure

Preparation and characterization of AAO Template of NiCrMo Nanowires

Aluminum sheet with thickness (0.5mm) was used for depositing the nanowires. Cleaning with acetone has been done for Al specimens and to get more active Al surface, the Al specimens were treated with 3M NaOH and then electrochemically polished also done. Oxalic acid with concentration 0.3 M was prepared in distilled water to use it as electrolyte for anodization. The anodization was achieved in two steps using Al specimen as an anode and stainless steel 316L as a cathode in an electrochemical cell with applying 30V.

Nickel sulfate 0.30 M, chromium sulfate 0.08 M, sodium molybdate 0.008 M and boric acid 30 g/L were used as electrolyte to deposit the NiCrMo nanowires. The AAO template was acted cathode in an electrochemical cell with Ni rode (purity 99.99%) acts as an anode. The voltage of deposition was 1.8 V for 40 min.

Scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) and Transition electron microscopy (TEM) were used for characterization of fabricated nanowires. These inspections were achieved for partial removal of the template after deposition of nanowires. The partial dissolving of AAO was performed by 10% HCl and ethanol.

Preparation and Characterization of Amalgam/Nanowires Composites

High-copper amalgam specimens were made by triturating 0.7 gm of powder alloy and the corresponding weight of mercury by amalgamator type (YDM-Pro) for 15 seconds at high speed with the composition of 56.7 Ag, 28.6 Sn and 14.7 wt% Cu. The specimens were prepared according to A.D.A. specification No.1; their dimensions were 1.5 cm in diameter and 5mm in height (cylinder specimen). For specimen preparation, a steel mould was used, after the triturating, the amalgam paste was immediately inserted into the cavity of the die with several thrusts of an amalgam condenser, and then a compressive stress has applied by inserting a punch in the die and applying $\approx$ 80 Kg/m^2 over the punch for 15 seconds. To prepare amalgam/nanowires composites, four weight percentages of nanowires (0.3, 0.6, 0.9 and 1.2 wt%) were replaced by a mixture of powder alloy in amalgam. Scanning electron microscopy (SEM) also used for characterization of fabricated composites.

Measurements of Properties

Atomic force microscopy (Veeco dinnova model) was used to observe the sample's surface in tapping mode, using cantilever with linear tips. The scanning area in the images was 5 µm × 5 µm and the scan rate was 0.6 HZ /second. In AFM examination, average roughness was measured for specimens.

The thermal conductivity of amalgam/nanowires composites was measured **by** Hot disk, TPS 500 obtained from Sweden. While microhardness was measured by HVS-1000 micro

hardness tester from LARYEE according to micro-indentation hardness principle ASTM E384 and ISO 6507 with a load of 9.8 N for 15 seconds. Averages then obtained from these measurements.

Results and Discussion

Characterization of Nanowires

Fig. (1) shows the SEM image and EDS analysis of fabricated NiCrMo nanowires, SEM image indicates growth of nanowires in plane view of partial removal for AAO. The growth of multilayer and the black and white color can be distinguished between the individual layers as a Ni matrix and formed phase, the fabricated wires are ordered with fixed length ranged between 420 and 500 nm, and this phenomenon confirms the homogenous growth of nanowires under experimental conditions as illustrated in TEM image of nanowires (Fig. 2). The EDS analysis of NiCrMo nanowires indicates obtaining 72.2 wt% Ni, 19.5 wt% Cr and 8.3 wt% Mo that close to the composition of known dental alloy (NiCrMo) and can be candidate these nanowires as a biomaterial.

TEM image of NiCrMo nanowires obtained after the partial dissolving of the template as arrays. This image shows the uniformly wire arrays with the same dimensions without distinguishing the phases in nanowires.

Characterization of Nanocomposites

In amalgam, the formed compounds are depended on particle shape (spherical or irregular) and on the chemical composition of the powders. Hg diffuses into powder particles and then reacts with Sn, Ag and Cu to form many compounds such (Sn–Hg, Ag–Hg and Ag–Cu) and Ag–Sn phases are remained from the reactants.

The microstructure of the dental amalgam is complex, consisting of new microphases, as produced in the reactions above, and the remains of the powder alloy particles, within the γ_1-Ag_2Hg_3 matrix phase [15].

The released Hg may react with Ag_3Sn to produce additional Sn_7Hg phase. The filling can lose its strength and becomes porous.

Figs. (3) to (7) show the SEM images of amalgam and amalgam/NiCrMo nanowires composites reinforced by adding four wt% include 0.3, 0.6, 0.9 and 1.2. The high copper alloy contains either all spherical particles or a mixture of irregular and spherical particles. The silver and tin particles are usually irregular, where as the Ag-Cu particles are usually spherical in shape. The admixed regular alloy has a composition close to the eutectic composition of Ag_3Cu_2. Fig. (3) indicates the admixed particular composition of amalgam without adding nanowires. The amalgamation reaction in admixed high-copper amalgam alloy is:

$$\gamma\text{-}Ag_3Sn + Ag\text{–}Cu_{(eutectic)} + Hg \rightarrow \gamma\text{-}Ag_3Sn + \gamma_1\text{-}Ag_2Hg_3 + \gamma_2\text{-}Sn_{7\text{-}8}Hg + Ag\text{–}Cu_{(eutectic)}\ \text{unreacted} \quad (1)$$

And the secondary or slow solid state reaction is:

$$\gamma_2\text{-}Sn_{7\text{-}8}Hg + Ag\text{–}Cu_{(eutectic)} \rightarrow \eta\,`Cu_6Sn_5 + \gamma_1\text{-}Ag_2Hg_3 + Ag\text{–}Cu \quad (2)$$

While the overall simplified reaction with Hg is:

$$\gamma\text{-}Ag_3Sn + \varepsilon\ Cu_3Sn + Hg \rightarrow \eta\,`\ Cu_6Sn_5 + \gamma_1\text{-}Ag_2Hg_3 \quad [15] \quad (3)$$

After adding nanowires, can be seen occurring more incorporation in composition of amalgam and reducing the difference in microstructure and disappearing the spherical particles, i.e. the reduction in unreacted γ. And there is a chance to react more Hg in amalgam because the nanowires behave as a network to collect the components in amalgam. By increasing the weight percentage of nanowires, the incorporation is increased and then the refining of the composition can occur as shown in Figs. (4) to (7) in addition to achieving a homogeneous distribution of phases and did not have any change in phase distribution by adding nanowires.

AFM Imaging

Figures (8) to (12) show the AFM images of conventional amalgam and its composites with nanowires. Each figure shows 2D and 3D images of the topography of material’s surface. It can be seen that the particle size and roughness were decreased after adding NiCrMo nanowires, this refining in surface leads to increasing regulation in structure. The addition of nanowires to amalgam gave lower roughness as shown in Table (1). Since there are many phases in amalgam lead to get surface irregularities, while after adding NiCrMo nanowires, many of vacancies may be filled and this can lead to getting smother surface. This result is confirmed to the refining which shown in SEM images.

At surface irregularities, attached bacteria can survive longer because they are protected against natural removal forces and oral hygiene measures [16], Moreover, roughening of the surface increases the area available for bacterial adhesion. The increasing of roughness leads to accumulation of plaque [17].

The bacteria adhesion forces to composite decrease with decreasing roughness of its surfaces. Also the surface roughness affects biofilm formation. As a restorative dental material, the lower roughness is favorite. Therefore, amalgam/nanowires composites are better than

conventional amalgam to reduce the accumulation of bacteria on their surface.

The increasing of smoothness confirms by the granularity accumulation distribution charts as shown in Fig. (13). It can be seen from Table (1) that the average diameter was decreased from 192.71 nm to 87.93 nm by adding NiCrMo nanowires, and this decreasing increases with increasing wires percent from 0.3 to 1.2 wt%.

Thermal Properties

The physical properties of dental amalgam are very important to evaluate the performance of this material. Thermal conductivity is an important problem in filling amalgam because it increases the sensitivity to the cold and hot foods and beverages. From Fig. (14) and Table (2) can be seen that the thermal conductivity and thermal diffusivity were decreased after adding NiCrMo nanowires for produced nanocomposites. This means that the transport of heat will be reduced during amalgam to the other parts in the mouth.

Electron transport in nanostructures is described by electrical G_E and thermal G_T conductance. Therefore, there are several analog between the two physical quantities. Electron transport in a nanowire causes two effects: electrical current and heat flux density. However, an analysis of thermal conductance is more complex than an analysis of electrical conductance because of the contribution of either phonons or electrons in the heat exchange. Quantized thermal conductance in one-dimensional systems has been predicted theoretically by Greiner [18] for the ballistic transport of electrons and by Rego[19] for the ballistic transport of phonons. Conductive channels are formed in one-dimensional systems. Each channel contributes to the total thermal conductance with the quantum of thermal conductance.

The lattice specific heat provides important information of the modified phonon spectrum in a low-dimensional system such as nanotubes and nanowires [20, 21]. The temperature dependent phonon mean-free-path is the result of scattering of phonons from domain boundaries, by defects, and/or phonon-phonon scattering [22].

The results suggest the dominance of phonon-boundary scattering, whereas the thermal properties of the more uniform and confined anisotropic NiCrMo nanowires demonstrate smooth temperature dependence, which suggests the dominance of phonon-phonon or phonon-defect scattering. These results suggest that the composite materials containing nanowires can be engineered for a wide range of applications. This result also an agreement with the observation of Nihar who studied the thermal properties of Cobalt nanowires [23]. On the other hand, Pierre et al. discussed the reduction in thermal conductivity of Ge and GaAs nanowires [24].

Microhardness

Mechanical properties of a material vary with the decrease in particle size. It is well known that the strength of metal and ceramic materials improves by decreasing grain size to nanosize or making a composite at the nanoscale. The results of measured microhardness for amalgam and its composites with nanowires are presented in Fig. (15).

This figure shows the increasing of microhardness with increasing wt% of added nanowires to amalgam. This result suggests the filling of vacancies in amalgam by nanowires and getting more coherent for components in amalgam especially with Hg. Therefore, can be seen the increasing in microhardness of amalgam/nanowires composites. The increasing of microhardness also observed by Noorhana et al. who used zinc oxide and aluminium oxide nanoparticles as fillers to enhance the hardness and other mechanical properties of dental amalgam material [13]. The results in present work are also good agreement with the observation which done by Bahremandi et al. who added 0.5, 1, 2 and 3 wt % of TiO_2 nanoparticles to improve the properties of amalgam [14].

Simulation Study

Finite element analysis (FEA) is a method that is being used more and more in industry to simulate structures and the loads that act on them. This method allows companies to foresee how a product will respond in real-time situations before they actually begin the construction of that product. As a result, they are able to alter their designs or materials using a limited number of prototype stages. Steady-state (static) thermal analysis in simulation has been done using two dimensional solid structures with 4-node and axisymmetric quadrilateral solid elements without extra shape functions as follow:

$$\mathbf{T} = \frac{\mathbf{1}}{\mathbf{4}}[\mathbf{T_1}(\mathbf{1} - \mathbf{s})] \quad (4)$$

where T is temperature at any x or y , T_1 is temperature of surrounding and s is shape function.

Temperature contour plots for pure teeth, amalgam and reinforced amalgam with NiCrMo nanowires are listed in Figs. (16) to (19). Four temperatures were selected to study thermal analysis of fabricated amalgams include 10, 20, 50 and 60 ºC (i.e., 283, 293, 323 and 333 K) as a temperature of circumstances (T_{inf}) compared with normal temperature of root equal to 37ºC (T_r). Amalgams in mouth are subjected to variation in temperatures according to hot and cold food and beverages and then stabilize at 37ºC (310K) which represents the temperature of normal saliva. This

study was achieved to determine the temperature distribution in the tooth and restorative materials.

In the presence of cold food and beverages in mouth (10 and 20 ºC), the amalgam/nanowires composites reach to steady-state (37ºC) more than conventional amalgam as shown in Figs. (16) and (17), i.e., the presence of NiCrMo nanowires within amalgam made it closed to natural teeth compared with conventional amalgam.

On the other hand, in the case of hot food and beverages in mouth (50 and 60 ºC), it was shown that the filling also reach steady-state for amalgam/nanowires composites more than for conventional amalgam as shown in Figs. (18) and (19). This result means that the distribution of temperature in reinforced amalgam proceed higher than in conventional amalgam and then the latter has a less chance to transfer the coldness and hotness to the other part of teeth through the filling.

Hot and cold conditions in the mouth create cyclic changes that could lead to thermal fatigue of the adhesive process. Compressive stresses are generated at the regions of load application, i.e., on the occlusal surface. Consequently, it is necessary to control this surface roughness by polishing it in order to prevent points of stress concentration and fatigue crack initiation, which can leads to fracture.

Simulation study is necessary to evaluate the effect of any new material as restorative filling. The results of thermal analysis indicated that there are improving in behavior of fabricated amalgam by adding NiCrMo nanowires through thermal conductivity caused by change temperature. These results are agreement with other simulated studies that achieved for dental materials, where Amit described thermo-mechanical finite element analysis in human tooth for determination of stress levels due to thermal and mechanical loads in healthy and restored tooth. Transient thermo-mechanical analysis simulating the ingestion of cold and hot drinks was performed to determine the temperature distribution in the tooth, followed by linear elastic stress analysis [25]. While Abdulsalm et al. investigated the influence of the cavity position on the stress values in the remaining tooth structure restored with amalgam or resin composite using Ansys 14 software [26]. Sunny et al. studied three different types of restorative material include Amalgam, gold alloy and composites using 3D model of mandibular premolar using solid works with a cavity in it by Ansys [27].

Conclusion

Reinforcing conventional amalgam was done by adding nanostructured material represented by NiCrMo nanowires with four weight percents (0.3, 0.6, 0.9 and 1.2) to improve some important properties. SEM images indicated the incorporation between amalgam's phases and added nanowires with getting refined surface as illustrated from AFM images in addition to get lower surface roughness. Thermal conductivity was measured for new nanocomposites and the results showed the decreasing in heat transfer through the amalgam reinforced with NiCrMo nanowires. Microhardness also tested to confirm the role of nanowires by increasing the microhardness due to remaining Hg within amalgam by masking of added nanowires.

Simulation study was achieved by Ansys 15 to get the theoretical study for effect the cold and hot foods and beverages on sensation of nanocomposites compared with conventional amalgam and the results candidate the better performance of nanocomposites.

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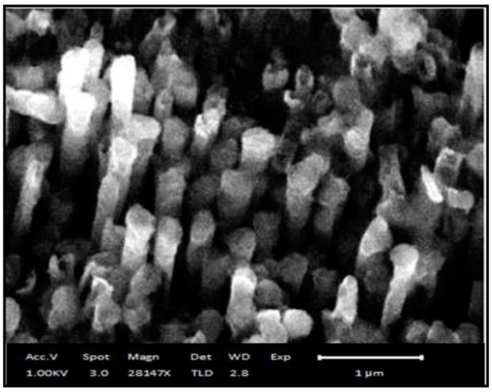
Table 1:Average diameter and roughness of specimens from AFM analysis.

Materials	Avg. diameter (nm)	Ra (nm)
Amalgam	192.71	18.1
Amalgam/0.3%nanowires	125.92	4.51
Amalgam/0.6%nanowires	112.16	3.45
Amalgam/0.9%nanowires	98.85	4.30
Amalgam/1.2%nanowires	87.93	2.52

Table 2: Thermal properties of amalgam and its composites.

Materials	Thermal conductivity (W/m.K)	Thermal diffusivity $x10^{-3}$ (mm^2/s)	Specific heat (MJ/m^3.K)
Amalgam	2.362	9.16	257.8
Amalgam/0.3%nanowires	1.845	5.99	308.0
Amalgam/0.6%nanowires	1.799	6.067	296.5
Amalgam/0.9%nanowires	1.797	6.050	297.0
Amalgam/1.2%nanowires	1.513	5.720	264.5



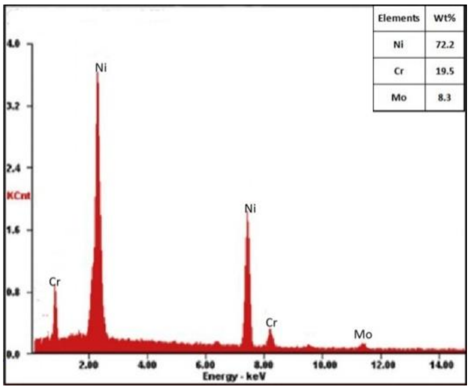


Figure 1: SEM image and EDS analysis of NiCrMo nanowires.

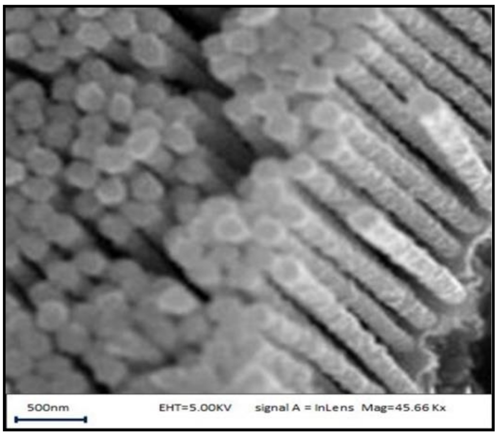


Figure 2: TEM images for NiCrMo NWs.

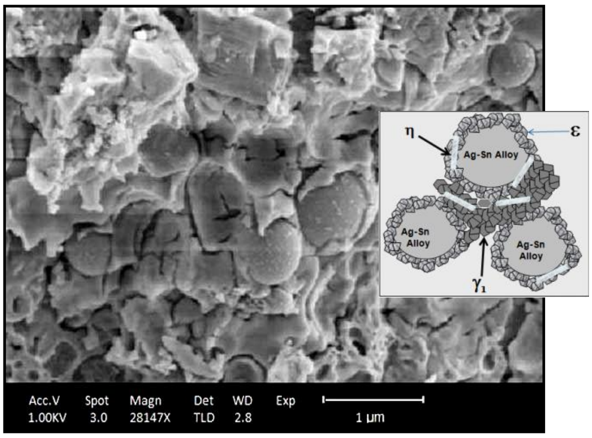


Figure 3: SEM image of amalgam.

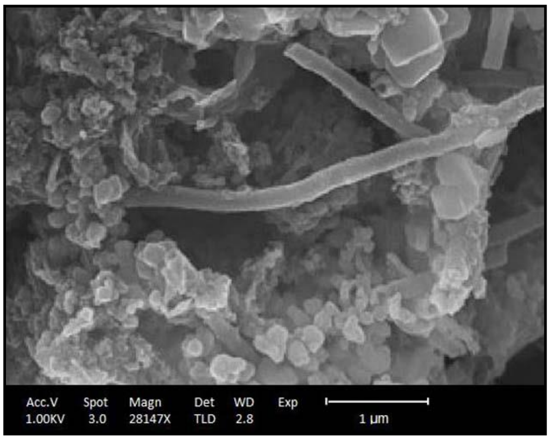


Figure 4: SEM image of amalgam/0.3NWs.

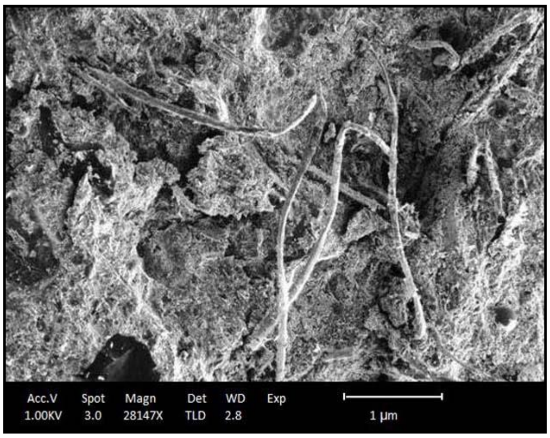


Figure 5 :SEM image of amalgam/0.6NWs.

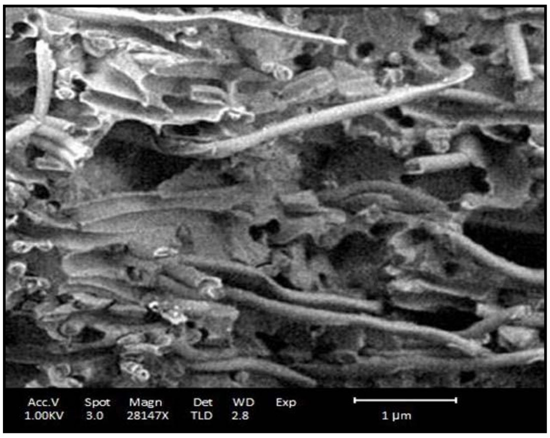


Figure 6: SEM image of amalgam/0.9NWs.

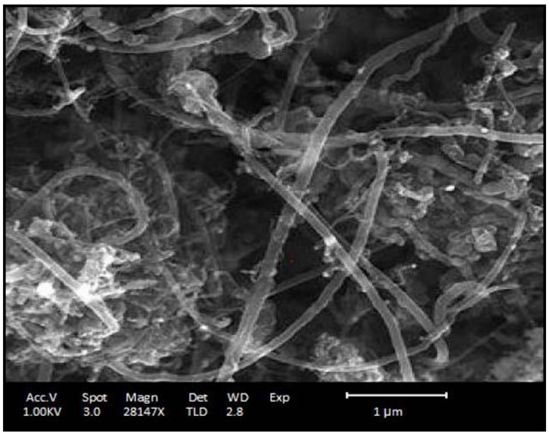


Figure 7: SEM image of amalgam/1.2NWs.

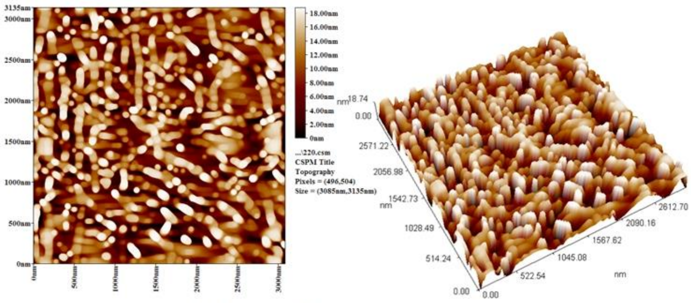


Fig. 11 AFM images of amalgam/0.9Nanowires.

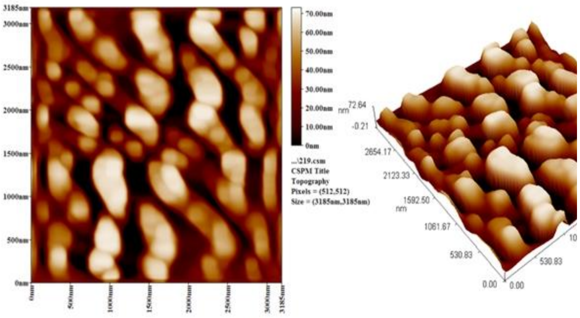


Fig. 8 AFM images of amalgam.

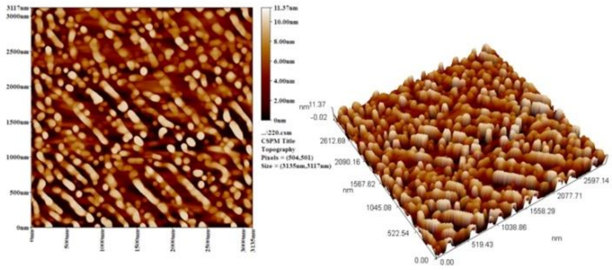


Fig. 12 AFM images of amalgam/1.2Nanowires.

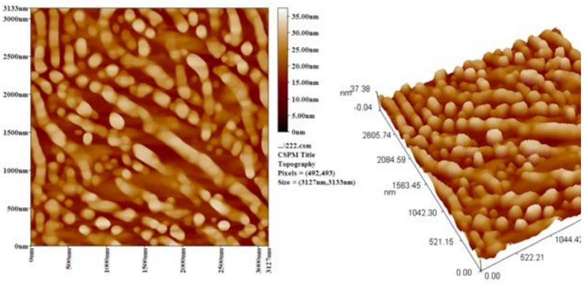
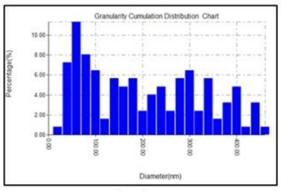
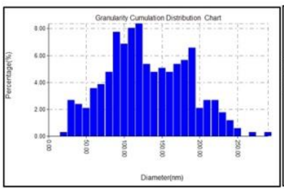


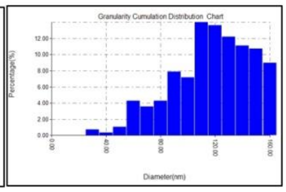
Fig. 9 AFM images of amalgam/0.3Nanowires.



Amalgam



Amalgam/0.3NWs



Amalgam/0.6NWs

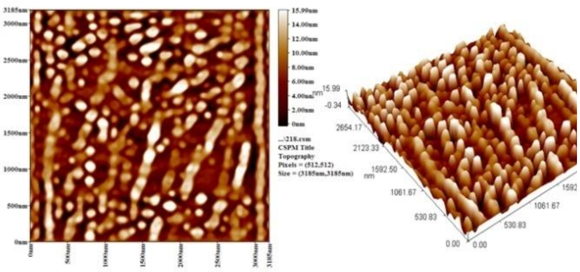
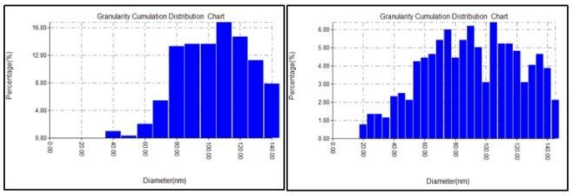


Fig. 10 AFM images of amalgam/0.6Nanowires.



Amalgam/0.9NWs Amalgam/1.2NWs

Fig. 13 Granularity accumulation distribution charts of amalgam and its composites.

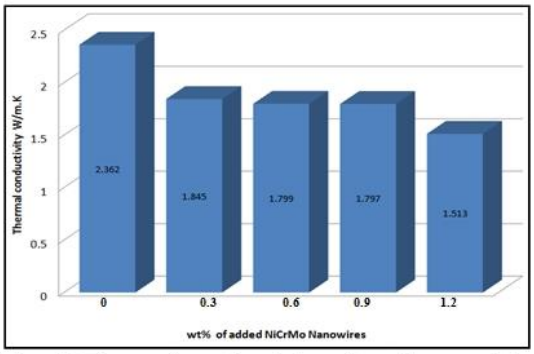


Fig. 14 Thermal conductivity of amalgam and its composites with NiCrMo nanowires.

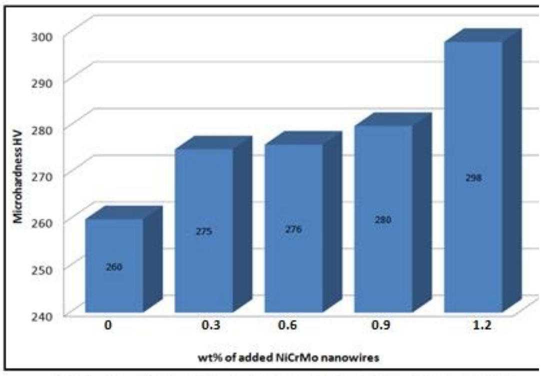


Fig. 15 Microhardness of amalgam and its composites with NiCrMo nanowires.

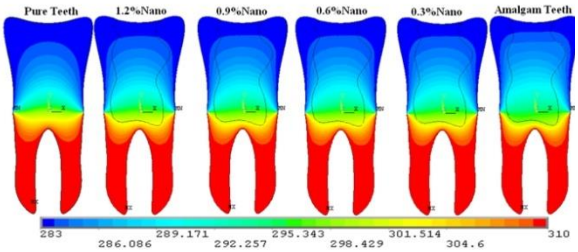


Fig. 16 Temperature contour plot at T_r=310 K and T_{inf}=283K.

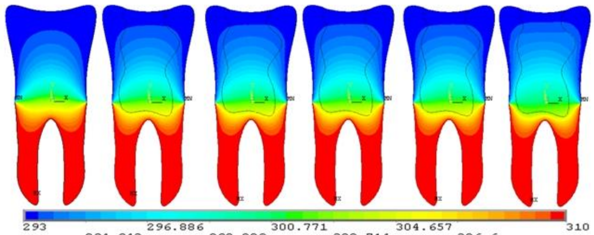


Fig. 17 Temperature contour plot at T_r=310 K and T_{inf}=293K.

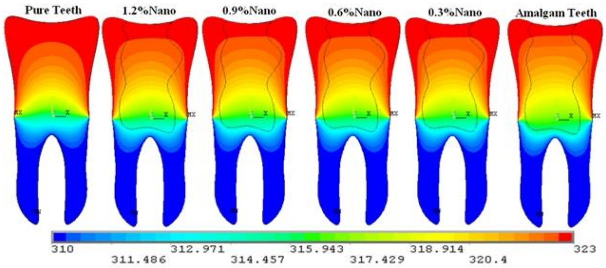


Fig. 18 Temperature contour plot at T_r=310 K and T_{inf}=323K.

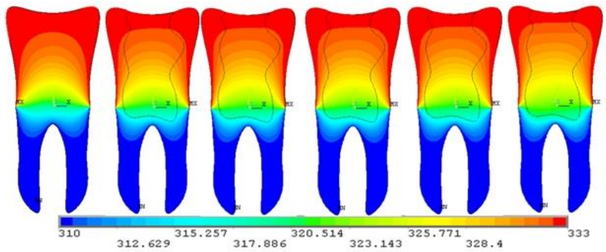


Fig. 19 Temperature contour plot at T_r=310 K and T_{inf}=333K.