Abstract

In this study NiTi based shape memory alloys were prepared using powder metallurgy technique. NiTi alloys as a reference alloy and NiTi with different additives of Cu have been produced individually.

Master samples were prepared using powder mixture of 55wt. %Ni and 45wt. % Ti, by mixing in electric mixture for 6 hours, then compacted at (150,250,350and450) MPa, and sintered under protected atmosphere of argon gas. It is concluded that the sintering at 850±5°C for 9 hours of all samples (without and with different addition of copper) are sufficient to satisfy sintering completely and to transform Ni, Ti and Cu into alloy structure .The same approach was made for the samples with (2.5, 5, 7.5and10) wt. % of Cu additions.

The results of X-ray diffraction showed that the master samples compacted at 450 MPa and sintered at 850±5°C for 9 hours consisted of three phases at room temperature(NiTi monoclinic phase, NiTi cubic phase and Ni_3Ti hexagonal phase).In addition to ($CuNi_2Ti$) intermetallic compound appeared in the samples with (2.5,5,7.5and10) wt.% of Cu additions. The increasing in compaction pressure from (150 to 450) MPa for all samples (without and with different additives of copper) resulted in an improvement in shape memory effect properties (SME) and mechanical properties of the prepared alloys .It also resulted in an decrement in porosity of the prepared alloys and decrement in the corrosion rate. Scanning electron microscope (SEM) observation indicated that most of the prepared alloys (without and with different additives of copper) compacted at (150 and 450) MPa and sintered at 850°C±5 for 9 hours have a clear martensitic structure. Differential scanning calorimeter (DSC) results demonstrate that the transformation temperature ranged from 26-100°C. This indicates that all samples at room temperature (30°C) consist of martensite phase.

The additions of Cu with (2.5-10) wt. % to NiTi have a significant effect on the prepared alloy, which resulted in an increment in the porosity and relatively a decrement in the hardness as compared to master samples. Also, there was distinguished increase in SME properties obtained from hardness test for the prepared NiTi alloy with addition of copper .The SME of master sample compacted at 450MPa are 5.85% compared with 5.982%, 7.33%, 8.061% and 7.194% for prepared NiTi with (2.5, 5, 7.5 and 10) wt. % Cu respectively compacted at 450 MPa. This proves that copper addition contributes in increasing the compressive strength (σcom), yield strength (σy) and modulus of elasticity (E) for NiTi alloys .The improvement percentage for 45wt.%Ni-45wt.%Ti-10wt.%Cu compacted at 450 MPa reached to 203% for (σcom)and 103% ,244% for σy and E respectively with comparison to 55wt.%Ni-45wt.%Ti sample compacted at 450 MPa .

A dense and uniform single Polymethylmethacrylate (PMMA) and composite Polymethylmethacrylate-Hydroxyapatite (PMMA/HA) coating layers were successfully deposited on the outer surface of the prepared alloys (without and with copper addition).SEM observation proved the good coverage of the

coating on the surface of NiTi samples results in giving good thick layer on the surface .Furthermore , there are no visible cracks or pores .

Corrosion behavior is conducted in synthetic saliva and Hank's solution by using open circuit potential (O.C.P) measurement and potentiostatic polarization test for uncoated samples and both coated (PMMA and PMMA/HA) samples. Also the polarization resistance (R_P) obtained from linear polarization a curve is studied for uncoated and coated samples.

The results of corrosion tests referred to decreasing in Icorr. and thus decreasing corrosion rate (CR) of prepared sample after additions of copper .The samples with(7.5)wt.% of Cu addition compacted at 150 MPa resulted in 97.02% an improvement in corrosion resistance in synthetic saliva and 96.28% in Hank's solution compared with master samples compacted at 150 MPa.

The general corrosion resistance in synthetic saliva and Hank's solution are better for PMMA/HA coating compared with PMMA coating and original sample without coating. The improvement in corrosion resistance for PMMA coated samples in synthetic saliva and Hank's solution for master samples compacted at 450 MPa are 98.82% and 99.69% respectively, compared to uncoated samples while for PMMA/HA coated samples are 99.98% in synthetic saliva and 99.99% in Hank's solution with compared to uncoated samples.

The polarization resistance R_P values in synthetic saliva and Hank's solution for uncoated master samples compacted at 450 MPa are 13.471×10^2 $\Omega.cm^{-2}$ and $3.570\times10^2\Omega.cm^{-2}$ respectively which are much lower than 258.636×10^2 $\Omega.cm^{-2}$and 53.426×10^2 $\Omega.cm^{-2}$ respectively for uncoated samples with 5wt. % Cu copper addition. Furthermore, a significant increase in R_P values in synthetic saliva and Hank's solution for samples compacted at 450 MPa with 7.5wt. % Cu coated with PMMA/HA reached to 292.381×10^6 $\Omega.cm^{-2}$ and 232.311×10^6 $\Omega.cm^{-2}$ respectively, compared to uncoated samples at the same condition which have R_P equal to 253.295×10^2 $\Omega.cm^{-2}$ in synthetic saliva and 219.602×10^2 $\Omega.cm^{-2}$ in Hank's solution .

The results of dissolution test demonstrate that the copper addition to NiTi with 7.5wt. % decrease the Ni ions released from samples to 0.14 ppm after 5 weeks immersion in synthetic saliva and 0.31PPm after 5weeks immersion in Hank's solution with compared to 0.78 ppm and 1.6 ppm for samples without copper after 5 weeks immersion in synthetic saliva and Hank's solution respectively. On the other hand for all coated samples, neither Ni nor Cu ions in synthetic saliva and Hank's solution were detected by AAS technique.

The biocompatibility of (PMMA and PMMA/HA) coated samples is better than that of the original NiTi without coatings.