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Research Article

Improvement in the Mechanical Properties of High Temperature Shape Memory Alloy (Ti₅₀Ni₂₅Pd₂₅) by Copper Addition

Saif ur Rehman,¹ Mushtaq Khan,¹ A. Nusair Khan,² Syed Husain Imran Jaffery,¹ Liaqat Ali,¹ and Aamir Mubashar¹

 ¹School of Mechanical and Manufacturing Engineering (SMME), National University of Science and Technology (NUST), Islamabad 44000, Pakistan
²Institute of Industrial Control System, Rawalpindi 46000, Pakistan

Correspondence should be addressed to Mushtaq Khan; mkhan_nust@yahoo.com

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High temperature shape memory alloys $Ti_{50}Ni_{25}Pd_{25}$ and $Ti_{50}Ni_{20}Pd_{25}Cu_5$ were developed, characterized, and tensile tested in both martensite ($M_f - 50^{\circ}C$) and austenite ($A_f + 50^{\circ}C$) phases. The transformation temperatures of ternary $Ti_{50}Ni_{25}Pd_{25}$ alloy were increased by 11 to 12.5°C by substitution of Ni with 5 at% Cu. At the same time, transformation heat absorbed and released during forward and reverse martensitic transformation was also increased. In the martensite phase, the mechanical properties, that is, the stress for reorientation of martensite variants and fracture stress, were increased by 33 and 60 MPa, respectively, whereas the fracture strain was decreased by 1.5%. In the austenite phase, the critical stress for slip and fracture stress were increased by 62 and 40.9 MPa, respectively, whereas the fracture strain was decreased by 1.2%. The increase in both stresses was attributed to the solid solution strengthening by substitution of Ni atoms with relatively greater atomic radius of copper (Cu) atoms. The overall results suggest that the addition of 5 at% Cu in place of Ni in $Ti_{50}Ni_{25}Pd_{25}$ alloy is very beneficial to improving the mechanical and shape memory properties and increasing the transformation temperatures.

1. Introduction

NiTi shape memory alloys are widely used in many engineering and medical fields due to their outstanding superelastic and shape memory properties [1]. Recently, the application of NiTi alloys has been extended in the industries like power generation, automotive, oil and gas exploration, and aerospace as solid state actuators [2–4]. However, in the mentioned applications, the actuators are to be operated at higher temperature due to high temperature environment (temperature greater than 100°C). Therefore, it is needed for the NiTi shape memory alloys to raise their phase transformation temperatures. The transformation temperatures of the NiTi base alloys have been successfully increased by alloying with some elements like Pd, Pt, Au, Zr, and Hf [5– 10]. However, alloying of Pd and Pt has got relatively more attention as compared to other ternary alloying elements, due to its comparable properties of high work output and good workability like NiTi alloys [11–14]. In addition to these properties, TiNiPd has narrow thermal hysteresis which is desired for fast and active control of actuators [11].

As the actuators are exposed to high temperature environment during operation, therefore, for reliable and long-life performance, it is necessary for the high temperature SMAs to have microstructural stability and resistance to oxidation at elevated temperatures. It should have enough strength in the martensite phase to resist transformation induced plasticity [15]. Apart from that, it should also have high critical stress for slip deformation to resist recovery, recrystallization, and creep in the high temperature austenite phase. The proposed techniques for strengthening the alloy against plastic deformation, thermal driven mechanisms and enhancing the dimensional stability include solid solution strengthening [16–19], precipitation hardening [20, 21], thermomechanical treatment [21], and annealing after cold working [22].

Alloying of different quaternary elements to TiNiPd has been thoroughly investigated by very few researchers [11, 16-19]. Solid solution strengthening has been carried out by addition of 0.12 and 0.2 at% boron in TiNiPd [16, 17]. It has been reported that the ductility of TiNiPd alloy at room and high temperature was improved due to formation of fine TiB₂ precipitates. Due to addition of boron, grain size of the alloy was refined and resulted in improved ultimate tensile strength; however, no improvement in the shape memory properties was observed. Substitution of 0.5 at% scandium with Ti in Ti_{50.5}Ni_{24.5}Pd₂₅ high temperature shape memory alloys has been reported by Atli et al. [11]. By addition of scandium, the transformation temperatures were decreased by 6° C in M_s and 10° C in M_f. The strength against irreversible deformation was observed to be improved as compared with ternary base alloy. Both the ternary Ti_{50.5}Ni_{24.5}Pd₂₅ and quaternary Ti₅₀Ni_{24.5}Pd₂₅Sc_{0.5} alloys exhibited very narrow thermal hysteresis as compared to NiTi binary alloys. The effects of addition of 1.0 at% scandium in place of Ti in Ti_{50.3}Ni_{24.7}Pd₂₅ on microstructure and transformation temperatures were also investigated by Ramaiah et al. [23]. It was reported that the transformation temperatures were significantly decreased by 42°C; M_f temperature of 181°C of ternary Ti_{50.3}Ni_{24.7}Pd₂₅ base alloy decreased to 139°C for quaternary $Ti_{49.3}Ni_{24.7}Pd_{25}Sc_{1.0}$ alloy. However, at the same time, thermal hysteresis also decreased from 15°C to 7°C. The effects of 5 and 10 at% copper addition in Ti₅₀Ni₂₅Pd₂₅ on the microstructure and shape memory properties were studied. It has been reported that strength of B19 martensite and transformation temperature are slightly increased but the thermal hysteresis remained the same. It was also noted that addition of Cu increases the dimensional stability of alloy at higher stress levels due to increasing the resistance against viscoplastic deformation by solid solution hardening mechanism [18]. In our previous work [21, 24], it has been shown that after thermomechanical training and precipitation hardening process thermal stability and recovery ratio were improved significantly in Ti₅₀Ni₁₅Pd₂₅Cu₁₀ alloys; however, the transformation temperatures were decreased.

To improve the shape memory properties of alloys, it is important to add such a solid solution strengthening agent that increases the yield stress in austenite phase (critical stress for slip) and decreases the yield stress (stress for reorientation of martensite variant) in martensite phase. By this way, if the difference between the critical stress for slip and stress required for reorientation is increased, the shape memory properties will be improved. However, for this purpose it is necessary to investigate the mechanical properties (critical stress for slip and stress required for reorientation of martensite variants) in austenite phase (above A_f temperature) and martensite phase (below M_f temperature), respectively. In the present study, the effect of substitution of Ni with 5 at% Cu in Ti₅₀Ni₂₅Pd₂₅ was investigated for low and high temperature mechanical properties and phase transformation temperatures.

2. Materials and Methods

Ti₅₀Ni₂₅Pd₂₅ and Ti₅₀Ni₂₀Pd₂₅Cu₅ high temperature shape memory alloys were prepared by vacuum arc melting process using tungsten electrode and water cooled copper crucible. High purity elemental constituents 99.98 wt.% Ti, 99.98 wt.% Ni, 99.99 wt.% Pd, and 99.99 wt.% Cu were used for preparation of these alloys. The cast buttons were melted 6 times and turned over after each melting cycle to ensure alloying homogeneity. The cast buttons were sealed in quartz tube after being vacuumed, filled with argon gas, and then homogenized at 950°C for 2 hours and water quenched. The homogenized buttons were sliced into 0.4 mm thick strips by wire electrical discharge machine (EDM). The 0.4 mm thick strips were cold rolled by 25% and their thickness was reduced to 0.3 mm. Samples for differential scanning calorimetry (DSC) and tensile testing were machined using wire EDM. All the samples were solution treated at 900°C for 1 hour and quenched in cold water without breaking the quartz tubes.

Phase transformation temperatures of both alloys in the solution treated condition were determined by DSC at a heating/cooling rate of 5°C/min under vacuum. The sample size for DSC analysis was kept as $2 \text{ mm} \times 2 \text{ mm} \times 0.3 \text{ mm}$. Isothermal tensile tests were performed to measure the mechanical behavior using tensile and compression testing machine equipped with MTS controller and software. Inhouse designed and manufactured holding grips were used to grip the samples. Load cell of 100 KN was used and strain measurement in tension was carried out by the movement of crosshead directly. Samples were heated by induction process and a K-type thermocouple was attached directly to the central portion of the samples by wrapping the copper wire for temperature measurement.

After gripping the sample and attaching thermocouple, each sample was heated at required isothermal test temperature while controlling load at 0 N. The samples were allowed to stay at isothermal test temperature for 5 minutes to stabilize the thermal fluctuations. The samples were strained at a strain rate of 1×10^{-4} mm/sec. For both alloys, only two isothermal tensile tests were performed, one at 50°C below M_f and one at 50°C above A_f.

3. Results and Discussion

3.1. Effect of Cu Addition on Transformation Temperatures. DSC analysis was carried out to investigate the effect of 5 at% Cu addition on phase transformation temperatures of Ti₅₀Ni₂₅Pd₂₅. Figure 1 shows the DSC heating and cooling cycles for Ti₅₀Ni₂₅Pd₂₅ and Ti₅₀Ni₂₀Pd₂₅Cu₅, with solution treated at 900°C for 1h. Phase transformation temperatures (M_s , martensite start; M_f , martensite finish; A_s , austenite start; and A_f , austenite finish) of both of the samples were measured from the intersection of the base line and the linear portions of exothermic or endothermic peaks as shown in Figure 1. The measured transformation temperatures of Figure 1 were summarized in Table 1 to compare the change in transformation temperatures for Ti₅₀Ni₂₀Pd₂₅Cu₅ with respect to the baseline Ti₅₀Ni₂₅Pd₂₅ alloy. Both alloys



FIGURE 1: DSC heating and cooling curves showing the transformation temperatures of $Ti_{50}Ni_{25}Pd_{25}$ and $Ti_{50}Ni_{20}Pd_{25}Cu_5$ alloys.

exhibited very similar behavior with single-stage martensite transformation and demonstrated proper developed peaks with well calculated transformation temperatures. However, the transformation heat (ΔH_c) released (area under cooling curve) during forward transformation cycle and transformation heat (ΔH_h) absorbed (area under heating curve) during reverse transformation cycle for Ti₅₀Ni₂₀Pd₂₅Cu₅ are greater than those of Ti₅₀Ni₂₅Pd₂₅Cu₅ alloy are 11 to 12.5°C and are higher than the ternary baseline Ti₅₀Ni₂₅Pd₂₅ alloy as shown in Table 1. The M_s temperature of Ti₅₀Ni₂₅Pd₂₅ increased by 12.5°C from 142.5°C to 155°C, while the A_f temperature increased by 11°C from 167°C to 178°C upon addition of 5 at% Cu in place of Ni.

From the above experimental results, it has been observed that substitution of Ni by 5 at% Cu increased the transformation temperatures and transformation heats significantly. The changes in transformation temperatures and transformation heats are actually due to the change in Ni/Pd content, because Cu content does not affect the transformation temperatures [25]. In the referred study, it has been reported that, according to the TiNi-TiPd pseudobinary phase diagram, the increase in Pd content and decrease in Ni content increase the transformation temperatures [26]. Moreover, according to Clausius-Clapeyron equation, the increase in transformation temperature causes increases in the transformation heats [26]. Thus, the increase in Ni/Pd ratio (as Ni content decreased and Pd content remained constant) resulted in increase in transformation temperatures and transformation heats absorbed and released during forward and reverse martensitic transformation, respectively. Moreover, it can also be noted from Table 1 that, by substitution of Ni with 5 at% Cu, the thermal hysteresis decreased by 1.5°C from 24.5°C to 23°C.

3.2. Effect of Cu Addition on Mechanical Properties. It is important to investigate the mechanical properties of shape memory alloys in both phases, that is, martensite and austenite, because the stress-strain relations in both phases are different from each other. Other conventional structural

TABLE 1: Transformation temperatures determined from DSC cooling and heating cycles of Figure 1 for $Ti_{50}Ni_{25}Pd_{25}$ and $Ti_{50}Ni_{20}Pd_{25}Cu_{5}$.

Alloy	Transformation temperatures (°C)				Thermal hysteresis
	M_s	M_f	A _s	A_f	$A_f - M_s$
Ti ₅₀ Ni ₂₅ Pd ₂₅	142.5	119	150.5	167	24.5
${\rm Ti}_{50}{\rm Ni}_{20}{\rm Pd}_{25}{\rm Cu}_5$	155	133	156	178	23

materials like steel have maximum yield strength at room temperature and then it decreases by increasing the testing temperature above 100°C. Conversely, for high temperature shape memory alloys, the yield strength at room temperature in martensite phase (lower than M_f) must be less than the yield strength at higher temperature (greater than A_f) in austenite phase. Austenite yield strength represents the critical stress for slip deformation while martensite yield strength represents critical stress for shear of martensite twins. For feasible actuators, the critical stress for shear must be lower than the critical stress for slip, so that when stress is applied, it results in shape deformation by shear of martensite twins rather than via dislocation generation and movement.

Tensile stress-strain curves tested in the martensite condition, 50°C below M_f for Ti₅₀Ni₂₅Pd₂₅ and Ti₅₀Ni₂₀Pd₂₅Cu₅ alloys, are shown in Figure 2. The yield stress for both alloys was calculated by drawing a parallel line, 0.2% offset to the elastic region of stress-strain curve as shown in Figure 2. The 0.2% yield stress (σ_{ν}) calculated from these curves in the martensitic condition for both alloys is shown in Table 2. This yield stress corresponds to the stress required for reorientation of martensite twins (also called stress for detwinned martensite). From Table 2, it can be observed that martensite yield stress of Ti₅₀Ni₂₀Pd₂₅Cu₅ was increased by 33 MPa with respect to the baseline Ti₅₀Ni₂₅Pd₂₅ alloy. Similarly, the stress at which the fracture occurred in the material (σ_f) also increased by 60 MPa; 951.6 MPa for $Ti_{50}Ni_{25}Pd_{25}$ was increased to 1011.6 MPa for Ti50Ni20Pd25Cu5. However, the strain at which the fracture occurred in the material (ε_f) was decreased by 0.75%; fracture strain of 10% for Ti₅₀Ni₂₅Pd₂₅ was decreased to 9.25% for $Ti_{50}Ni_{20}Pd_{25}Cu_5$.

Figure 3 represents the tensile stress-strain curves tested in the austenite condition, 50°C above A_f for $Ti_{50}Ni_{25}Pd_{25}$ and Ti₅₀Ni₂₀Pd₂₅Cu₅ alloys. Measurement of austenite yield strength was carried out by the same procedure as discussed earlier and as shown in Figure 3. The 0.2% yield stress (σ_{ν}) calculated from these curves in the austenitic condition for both alloys is shown in Table 2. This yield stress represents the critical stress for slip; stress required for the deformation of material through dislocations generation and their movement. The austenite yield stress of $440\,\text{MPa}$ for $\text{Ti}_{50}\text{Ni}_{25}\text{Pd}_{25}$ was increased to $502\,\text{MPa}$ for Ti₅₀Ni₂₀Pd₂₅Cu₅ and this resulted in net increase of 62 MPa as shown in Table 2. The fracture stress (σ_f) also increased by 40.9 MPa; 1121.3 MPa for Ti₅₀Ni₂₅Pd₂₅ was increased to 1162.2 MPa for $Ti_{50}Ni_{20}Pd_{25}Cu_5$. Here, the fracture strain (ε_f) was observed to be decreased by 1.2%; fracture strain of 8.5% for Ti₅₀Ni₂₅Pd₂₅ was decreased to 7.3% for Ti₅₀Ni₂₀Pd₂₅Cu₅.



FIGURE 2: Tensile stress-strain curves tested in the martensite condition, 50° C below M_f of (a) $Ti_{50}Ni_{25}Pd_{25}$ and (b) $Ti_{50}Ni_{20}Pd_{25}Cu_5$ alloys.

TABLE 2: Yield stress, fracture stress, and fracture strain calculated from stress-strain curves of Figures 2 and 3 for $Ti_{50}Ni_{25}Pd_{25}$ and $Ti_{50}Ni_{20}Pd_{25}Cu_5$ alloys.

Alloy	Phase	Testing temperature (°C)	0.2% Yield stress, σ_y (MPa)	Fracture stress, σ_f (MPa)	Fracture strain, ε_f (%)
Ti ₅₀ Ni ₂₅ Pd ₂₅	Martensite	69	290	951.6	10
	Austenite	217	440	1121.3	8.5
$Ti_{50}Ni_{20}Pd_{25}Cu_5$	Martensite	83	323	1011.6	9.25
	Austenite	228	502	1162.2	7.3

From the experimental results shown above, it can be observed that, by substitution of 5 at% Ni by 5 at% Cu, the yield stress and fracture stress in the martensite phase were increased by 33 and 60 MPa, respectively, whereas the fracture strain was decreased by 1.5%. Similarly the yield stress and fracture stress in the austenite phase were increased by 62 and 40.9 MPa, respectively, whereas the fracture strain was decreased by 1.2%. The increase in both vield stresses (stress for reorientation of martensite and stress for slip deformation) and fracture stress (maximum stress) is attributed to the solid solution strengthening due to partial substitution of Ni with Cu. Here, the atomic radius of Cu (0.128 nm) is relatively greater than the atomic radius of Ni (0.125 nm) and is therefore responsible for solid solution strengthening. By solution strengthening effect, the ductility of Ti₅₀Ni₂₀Pd₂₅Cu₅ was lowered and resulted in relatively low fracture strain in both phases.

It can be observed that the increase in stress for reorientation of martensite variants (although it is not required for better shape memory alloys) was observed to be 33 MPa. On the other hand, the critical stress for slip deformation (essentially required for better shape memory alloys) was increased by 62 MPa (Table 2). As the increase in critical stress for slip is more beneficial than the increase in stress for reorientation of martensite variants, it can be suggested that, by partial substitution of 5 at% Ni with 5 at% Cu, the critical stress for slip was increased by 29 MPa (difference in net increase in critical stress for slip and net increase in the stress for reorientation of martensite variants).

Figure 4 represents the tensile stress-strain curves (loading and unloading for 5 complete cycles) tested in the austenite condition, 50°C above A_f of $Ti_{50}Ni_{25}Pd_{25}$ and Ti₅₀Ni₂₀Pd₂₅Cu₅ alloys. For both alloys, the samples were first stressed continuously until the strain reached at 5% and were then unloaded completely. This process was repeated five times for both alloys. It can be observed that both alloys resulted in partial pseudoelasticity. The maximum stress 676 MPa reached at 5% strain for Ti₅₀Ni₂₅Pd₂₅ in the first loading cycle is lower than the stress 802 MPa reached at 5% strain for Ti50Ni20Pd25Cu5. Moreover, the stress hystereses that resulted in the first, third, and fifth cycles for Ti₅₀Ni₂₅Pd₂₅ alloy are 236, 210, and 190 MPa, respectively. Similarly, the stress hystereses obtained in the first, third, and fifth cycles for Ti₅₀Ni₂₀Pd₂₅Cu₅ alloy are 182, 160, and 160 MPa, respectively. Comparing the stress hysteresis of both alloys, it can be observed that the stress hystereses for Ti₅₀Ni₂₅Pd₂₅ were larger and continuously decreased as the number of cycles increased, whereas the stress hystereses for Ti₅₀Ni₂₀Pd₂₅Cu₅ were smaller and remained stable. Thus, it can be concluded that, by substitution of Ni by 5 at% Cu in Ti₅₀Ni₂₅Pd₂₅ alloy, the stress hysteresis was reduced and became stable.



FIGURE 3: Tensile stress-strain curves tested in the austenite condition, 50° C above A_f of (a) $Ti_{50}Ni_{25}Pd_{25}$ and (b) $Ti_{50}Ni_{20}Pd_{25}Cu_5$ alloys.



FIGURE 4: Tensile stress-strain curves (loading and unloading) tested in the austenite condition, 50°C above A_f of (a) $Ti_{50}Ni_{25}Pd_{25}$ and (b) $Ti_{50}Ni_{20}Pd_{25}Cu_5$ alloys. "*N*" represents number of cycles.

4. Conclusions

High temperature shape memory alloys Ti₅₀Ni₂₅Pd₂₅ and Ti₅₀Ni₂₀Pd₂₅Cu₅ were developed and characterized for measurement of phase transformation temperatures. They were tensile tested to investigate their mechanical properties in both martensite (M_f - 50°C) and austenite (A_f + 50°C) phases. The important conclusions drawn are reported as follows.

 The transformation temperatures of ternary Ti₅₀Ni₂₅Pd₂₅ alloy were increased by 11 to 12.5°C by substitution of Ni with 5 at% Cu; M_s temperature of Ti₅₀Ni₂₅Pd₂₅ increased by 12.5°C from 142.5°C to 155°C, while the A_f temperature increased by 11°C from 167°C to 178°C. The increase in transformation temperatures was attributed to the decrease of Ni content and increase of Ni/Pd content in the matrix.

- (2) The transformation heat absorbed and released during forward and reverse martensitic transformation was increased by substitution of Ni with 5 at% Cu.
- (3) In the martensite phase, the stress for reorientation of martensite variants and fracture stress were increased by 33 and 60 MPa, respectively, whereas the fracture strain was decreased by 0.75%. In the austenite phase,

the critical stress for slip and fracture stress were increased by 62 and 40.9 MPa, respectively, whereas the fracture strain was decreased by 1.2%, showing the improvement in mechanical properties.

- (4) The increase in both stresses was attributed to the solid solution strengthening by substitution of Ni atoms with relatively greater atomic radius of Cu atoms.
- (5) The relatively larger increase in the critical stress for slip by substitution of Ni with 5 at% Cu suggested that the shape memory properties were improved.
- (6) By substitution of Ni by 5 at% Cu in $Ti_{50}Ni_{25}Pd_{25}$ alloy, the stress hysteresis was reduced and became stable.

The overall results suggested that addition of 5 at% Cu in place of Ni in $Ti_{50}Ni_{25}Pd_{25}$ alloy is very beneficial to improving the mechanical and shape memory properties of the alloy. It also increased the transformation temperatures of alloy to a reasonable level.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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