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Research Paper

EFFECT OF AGING AND ALLOYING ON WEAR CHARACTERISTICS OF NITINOL BASED ALLOYS

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NiTiNOL is an alloy of roughly 50% Nickel and 50% Titanium. NiTiNOL derives its name from its chemical components and its founders: Ni (Nickel) + Ti (Titanium) + NOL (Naval Ordinance Lab). It has good biocompatibility and good magnetic resonance imaging opacity, making it ideally suited for design of biomedical implantable. In the present work, alloy samples with varying percentage of ternary alloys are vacuum cast in vacuum arc melting furnace and the alloy samples are heat-treated at different low temperatures keeping the time (1hour) constant and tested for hardness wear behavior of the samples. The wear characteristics are predicted for as cast samples as well as heat treated samples by carrying out Adhesive wear as well as Abrasive wear tests as per ASTM Standards. The hardness seems to increase with different aging temperatures (oC) conditions. The Vickers hardness is low for the as-cast samples of both the compositions but gradually increases with increase in the heat-treatment temperatures up to 350oC possibly due to formation of transition precipitates but there is a decrease in the hardness at 400oC possibly because of NiTi formation. Abrasive specific wear rates show a trend that is exactly the inverse of Vickers hardness. The wear rates tend to decrease from as-cast condition up to 350oC heat-treatment, and increase in the samples heat-treated at 400oC. The Vickers hardness of the samples is seen to go up gradually with an increase in the aluminum content, also the effect of heat –treatment will reduce the internal stresses and eliminate coring. The marginal hardness increase may be due to the Ni-Ti composition moving towards stoichiometry by precipitation of excess Ni or Ti in the form of suitable precipitates. Abrasive wear of the specimens is seen to increase both with an increase with the cooper content and the heat-treatment temperature, for different reasons. The specific wear rate for the samples is seen to go up accordingly with the abrasive wear since the ductility of the samples may be lowered considerably with an increased addition of aluminum to the NiTi matrix.

Keywords: Adhesive Wear, Abrasive Wear, Vickers Hardness, Heat-treatment

INTRODUCTION

Nickel titanium, also known as NiTiNol, is a metal alloy of nickel and titanium, where the two elements are present in roughly equal atomic percentages.

NiTiNol alloys exhibit two closely related and unique properties: shape memory and super elasticity (also called pseudo elasticity). Shape memory refers to the ability of NiTiNol to undergo deformation at one tem-

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perature, and then recover its original, undeformed shape upon heating above its "transformation temperature". Super elasticity occurs at a narrow temperature range just above its transformation temperature; in this case, no heating is necessary to cause the unreformed shape to recover, and the material exhibits enormous elasticity, some 10-30 times that of ordinary metal.

NiTiNol is an alloy of roughly 50% Nickel and 50% Titanium. NiTiNol derives its name from its chemical components and its founders: Ni (Nickel) + Ti (Titanium) + NOL (Naval Ordinance Lab).It has good biocompatibility and good magnetic resonance imaging opacity, making it ideally suited for design of biomedical implantable devices. More importantly are two characteristics that make NiTiNol extra ordinarily unique from other bioengineering metallic alloys, namely its super elasticity and shape-memory, which are described in further. These two unique properties are attributed to a first-order phase transformation from the parent austenite phase to the daughter marten site phase which can occur via the addition of an energy source either in the form of heat or stress. Nitinol has been used for a variety of applications: pipe couplings, bra underwires, earthquake dampeners, eyeglass frames, orthodontic wires, mobile phone antennas, micro-actuators, and a variety of biomedical devices. Recently, however, the NiTiNol community's trend has been to turn its efforts to the biomedical device field.

SHAPE MEMORY ALLOYS (SMAS)

Smart materials are a class of materials that exhibits a coupling between multiple physical domains. Common examples of smart materials include those that convert electrical signals into mechanical deformation and vice versa. Smart materials incorporated into a system are called a smart system that utilizes the coupling properties of smart materials to provide functionality.

Table 1: Shape Memory Alloys Commonly Used

Alloy	NiTi	CuZnAl	CuAlNi
Property			
Melting point (co)	1300	950-1020	1000-1050
Density (g/cm3)	6.45	7.64	7.12
Electrical Resistivity (μ Ω cm)	70-100	8.5-9.7	11.0-13.0
Thermal Conductivity, RT (W/m K)	18	120	30-43
Young's Modulus (GPa)	83 (austenite)	72 (beta phase)	85 (beta phase)
	26-48 (marten site)	70 (marten site)	80 (marten site)
Yield Strength (MPa)	195-690 (austenite)	350 (beta phase)	400 (beta phase)
	70-140 (marten site)	80 (marten site)	130 (marten site)
Ultimate tensile strength (MPa)	895	600	500-800
Shape memory strain (%)	8.5	4	4
Transformation range (o C)	<110	<120	<200
Transformation hysteresis (o C)	30 – 50	15 – 25	15 – 20

Over the last ten to twenty years, a number of materials have been given the term smart based on their interesting materials properties. Some of these materials exhibit a volume change when subjected to an external stimulus such as an electric potential, some shrink or expand when heated or cooled whereas are materials that produce electrical signals when bent or stretched.

Based on their properties smart materials are classified into the following categories:-

1. Piezoelectric materials - These are a class of materials that convert energy between mechanical and electrical domain.
2. Shape memory alloys - These refer to second class of smart material that is thermo-mechanical materials which deform when heated a cooled.
3. Electro active polymers- This class of materials exhibits electromechanical coupling. These are functionally similar to piezoelectric materials but exhibit much different electro mechanical response characteristics Final Stage

If the SMA encounters any resistance during this transformation, it cans extremely large forces. This phenomenon provides a unique mechanism for remote actuation.

The below table 1.1 shows shape memory alloys commonly used.

It is seen from the table 1.1 that are three classes of shape memory alloys, wherein NiTi has got the lowest density that gives it an added advantage in terms of lightweight and possesses a high value of shape memory strain of about 8%.

The major applications for these alloys are in the field of medicine and orthodontics with a few other areas of significance such as eyeglass frames, cellular phones antennas, and automotive devices.

Some of the applications are given below:

1. Aircraft
2. Micro-gripper
3. Eye glass frames
4. Industrial robot
5. Endovascular stent
6. Endodontic

NiTiNol alloys exhibits two closely related and unique properties: shape memory and super elasticity (also called pseudo elasticity). Shape memory refers to the ability of NiTiNol to undergo deformation at one temperature, and then recover its original, undeformed shape upon heating above its transformation temperature. Superelasticity occurs at a narrow temperature range just above At temperature; in this case, no heating is necessary to cause the undeformed shape to recover, and the material exhibits enormous elasticity, some 10-30 times that of an ordinary metal. Nitinol's extraordinary ability to accommodate large strains, coupled with its Physiological and chemical compatibility with the human body has made it one of the most commonly used materials in medical device engineering and design.

EXPERIMENTAL DETAILS

Alloy Composition

Choosing the composition of the alloy with varying Aluminium contents.

Table 2: Selected Alloy Composition (atomic %) for Experimental Work

	Ni	Ti	Al	Vd	Total
Alloy	Wt(gms)	Wt(gms)	Wt(gms)	Wt(gms	Wt(gms)
NiTi	55.076	44.932	0	0	100
NiTi Al,Vd	46.16	49.78	2.09	1.97	100

To weigh out the materials conversion from at (%) to wt (%) is done by using following relation.

$$\text{Wt\%A} = \frac{\text{atom\% A x at wt A}}{\text{(Atom \% A x at wt A) + (Atom \% B x at wt B) + (Atom)}}$$

Table 3: Atomic Mass of the Raw Materials (Ti, Ni, Al and Vd)

Ni	58.69
Ti	47.88
Al	26.98
Vd	50.94

Table 4: Alloy Composition wt (%) Selected for Experimental Work

	Ni	Ti	Al	Vd
Alloy	Wt (%)	Wt (%)	Wt (%)	Wt (%)
NiTi	55.076	44.932	0	0
NiTiAlVd	46.16	49.78	2.09	1.97

MELTING AND CASTING OF ALLOY

Melting of the alloy is carried out in the Vacuum arc-melting furnace. The pure metal rods (Ni, Ti, Al and VD) were cut into very small bits, weighed using an electronic balance of 0.001 grams accuracy and then charged into furnace for button melting operation. Vacuum is created using the standard procedure up to 10-5 mbar and ARGON gas is purged into chamber. The gas is flushed out by the rough pumping the system and the procedure for vacuum creation is carried out to achieve an ultimate vacuum of 10-5 mbar and then followed by the backfilling of ARGON gas. This is done in order to remove any impurities present in the chamber. The melting machine is put ON and melting of the four ingots is carried out to form buttons. The melting and remelting of the buttons is done a few times in order to achieve homogeneity of the alloy compositions. After achieving the homogeneity, the set-up is changed for the suction (rod drawing) operation and a button is charged into the copper crucible for melting. Vacuum is created using the same standard procedure up to 10-5 mbar; argon gas is purged followed by flushing it out and re-creation of vacuum with the same procedure followed by again by back filling of Argon gas. The suction and drawing of the rod is carried out after string and arc to render the alloy to molten form. An alloy rod of 3mm diameter and 60mm length forms by suction and quick cooling of the molten metal into crucible. Four rods were melted and cast in the melting furnace for each of the two composionsNiTi, NiTiAl, VD.

Table 5: Final Composition Selected for Experimental Work

	Ni	Ti	Al	Vd
Alloy	At (%)	At (%)	At (%)	At (%)
NiTi	50	50	0	0
NiTiAl, Vd	50	44	4	2

AGING OF THE ALLOYS

The alloy samples are heat-treated at different low temperatures keeping the time (1hour) constant. This is done in order to manipulate the transformation temperatures (At) of the alloys to achieve both super elastic and shape memory properties needed for medical applications such as orthodontic wires stints etc and medical appliances.

The table below shows the temperatures at which samples are aged.

Table 6: Heat Treatment Temperatures for the Alloy Samples

Alloy	Aging Temperatures(^{o}c)		
	300	350	400
NiTi	1sample	1sample	1sample
NiTiAlV	1sample	1sample	1sample

Aging treatment is given for the alloy samples in an INDFURR LTD furnace with ARGON atmosphere since titanium is highly pruned to oxidation when heated to high temperatures (>600.c). The alloy rods are charged into the furnace in groups for each heat treatment. The samples are heated at a constant rate of 10. Per minute to the required temperature and allowed to remain for the specified period of one hour and then air-cooled to room temperature. The influences of aging process on the properties of the alloys are studied by performing test such as microstructure, wear etc.

OPTICAL MICROSCOPY

Samples of small size approximately 5mm thickness are used for micro structural examination under optical microscopy. Samples were mounted using cold mounting acrylic resin and allowed to set. The surface preparation was done by successively grinding on 1/0, 2/0, 3/0 and4/0 emery papers for getting a flat surface. Further ground samples was polished on velvet cloth using alumina suspension and final polish was given using diamond paste on special velvet cloth meant for diamond polish to obtain a mirror finish.

Etching was carried out on the samples with the following Keller's enchant.

2ml HF +10ml HN03 +20ml H20

The surface of the specimen swabbed for a few seconds until shinning surface turns to dull finish. The specimen is immediately washed with water, then surface treated with a drop or two ethanol and then dried. The sample is placed on specimen support of the optical microscope (Dew inters) and structures studied at suitable magnifications and images grabbed.The same procedure is repeated for the other samples to view and capture the microstructure.

RESULTS AND DISCUSSION

Optical Microscophy

The optical micrographs of NITONOL and NiTiAlV in the as-cast and heat-treated conditions are shown below.

Figure 1: 1NiTi heat treated (300°C)

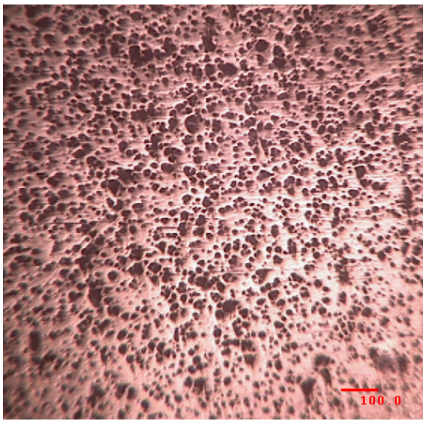


Figure 2: NiTi heat treated (350°C)

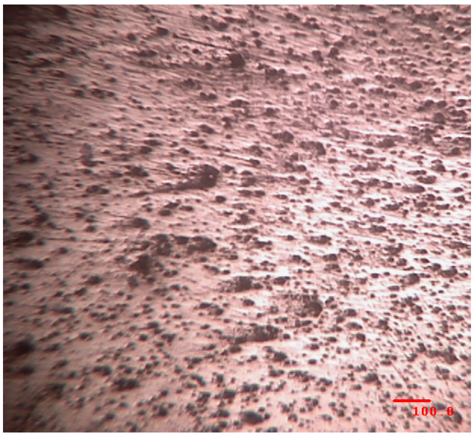


Figure 3: NiTi heat treated (400°C)

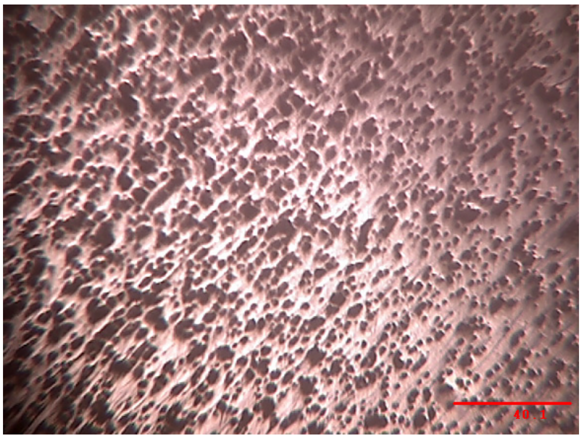


Figure 4: NiTiAlV as-cast Condition

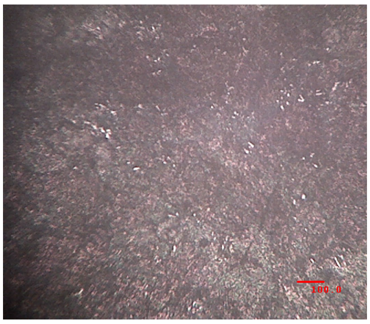


Figure 5: NiTiAlV Heat Treated (300°C)

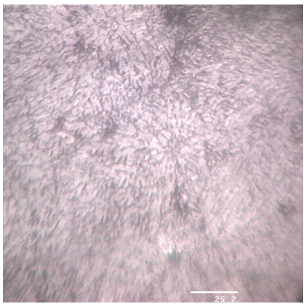


Figure 6: NiTiAlV Heat Treated (350°C)

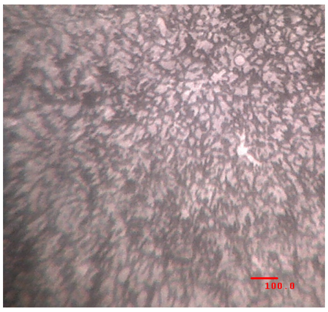
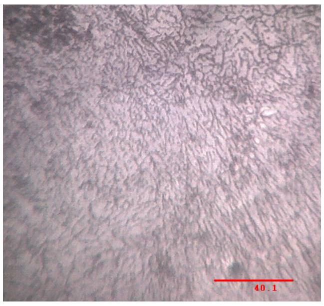


Figure 7: NiTiAlV Heat Treated (400°C)



Vickers Hardness

The variation of Vickers hardness for each of the as-cast and heat-treated conditions is tabulated below keeping the load constant as 5 kg on Vickers hardness machine.

Table 7: Vickers Hardness Variation for the Samples

	Specimen	Load(kg)	VHN(mean)
As cast	NiTi	5	451
	NiTiAlV	5	340
HT-300°C	NiTi	5	510
	NiTiAlV	5	470
HT-350°C	NiTi	5	540
	NiTiAlV	5	524
HT-400°C	NiTi	5	442
	NiTiAlV	5	458

The variation of Vickers hardness number for both the as-cast and the heat-treated condition of all the different compositions is shown graphically below. The hardness seems to increase with different aging temperatures (°C) conditions.

Figure 8: Vickers Hardness Number for NiTi for all Aging Conditions

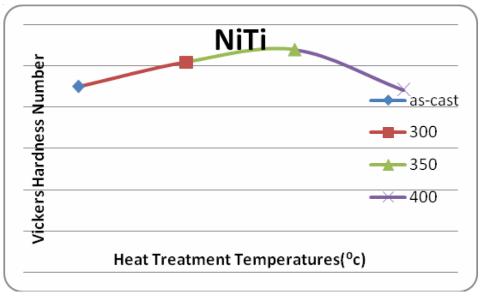
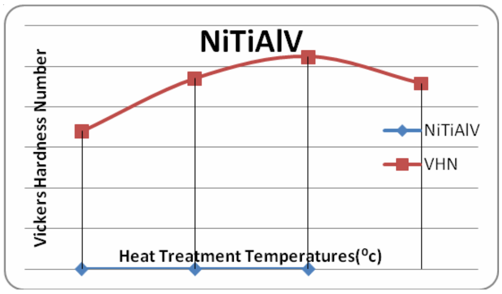


Figure 9: Vickers Hardness Number for NiTiAlV for all Aging Conditions



It is seen from the above graphs that the hardness of NiTiNol increases with the increase in aluminum content. The hardness of a NiTiNol does not change very much at the aging temperatures and times used here. The process of aging in three sets of samples seems to be similar.

WEAR

Adhesive Wear

Adhesive wear was carried out on the entire sample as per ASTM_99-95a. The three mm diameter specimens were tested at 1 and 2 kg loads. The wear in them were 1 to 5 microns only. With the list count in testing unit being a micron meaningful conclusion are difficult to draw. The results are to be anticipated as the NiTi's are hard and work hardened very rapidly. The wear will after every test reveals a golden colored smear indicating formation of TiO_2 coat from oxidation of Ti from the samples as shown in the figure below.

Figure 10: Aging Conditions

Hence it was thought better to get relative evaluation of the alloys by abrasive wear test.

Further Nitinol's are increasing used as endodontic tools for root canal treatment where they are to meet mild to strong abrasive environment abrasives wear testing looks relevant. Abrasive wear was carried out according to ASTM-132.

Abrasive Wear

Abrasive wear for the two compositions is carried out for both the as-cast and heat-treated samples tested for two loads using pin-on-disc apparatus fitted with an AA60 alumina (60µ) grinding wheel wheel from M/s carborundum.

The results for the three NiTi compositions is tabulated below.

Table 8: Wear Results for NiTi Compositions Under Different Loading Conditions

	Load(kg)	Speed(rpm)	Time(sec)	Wear(microns)
NiTi-as-cast	0.5	200	590	90
	1	200	585	158
NiTi-HT 300	0.5	200	585	81
	1	200	587	159
NiTi-HT 350	0.5	200	595	40
	1	200	590	165
NiTi-HT 400	0.5	200	545	84
	1	200	592	162

Table 9: Wear Results for NiTiAlV Compositions Under Different Loading Conditions

	Load(kg)	Speed(rpm)	Time(sec)	Wear(microns)
NiTi-as-cast	0.5	200	590	90
	1	200	585	158
NiTi-HT 300	0.5	200	585	81
	1	200	587	159
NiTi-HT 350	0.5	200	595	40
	1	200	590	165
NiTi-HT 400	0.5	200	545	84
	1	200	592	162

Figure 11: Wear Graph NiTi (0.5kg) Obtained from Experiment

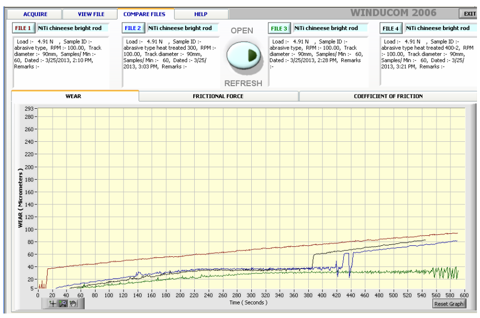


Figure 12: Wear Graph NiTi (1kg) Obtained from Experiment

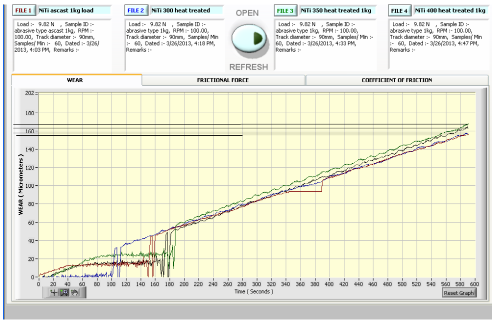


Figure 13: Wear Graph NiTiAlV (0.5kg) Obtained from Experiment

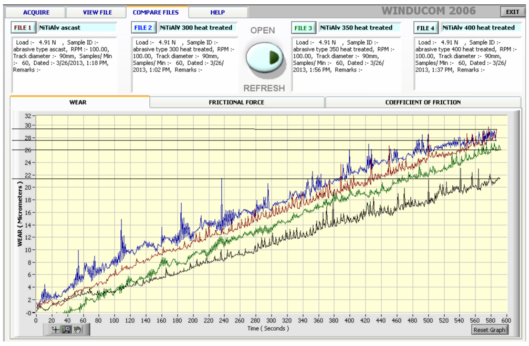
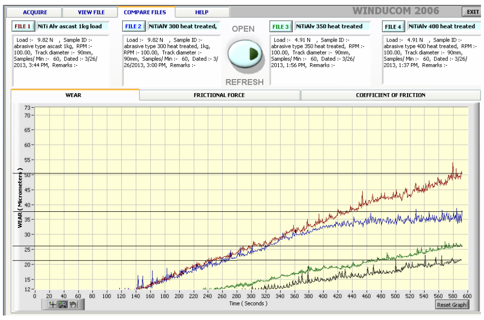


Figure 14: Wear Graph NiTiAlV (1kg) Obtained from Experiment



The above graph shows the wear of the two samples for 0.5 kg and 1 kg load application. It is observed that all two materials show similar wear behavior.

The wear results are graphically represented here under

Figure 15: Wear of as-cast and Heat Treatment Specimens of NiTiAlV for Both Loading Conditions

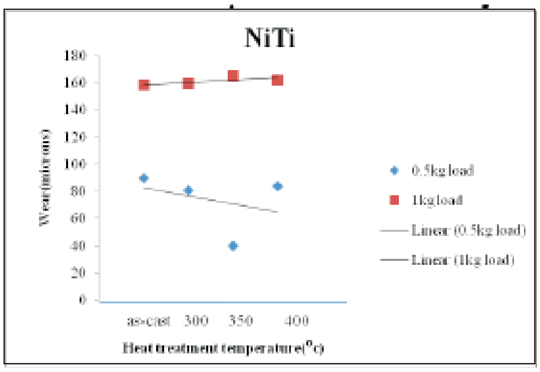


Fig (a)

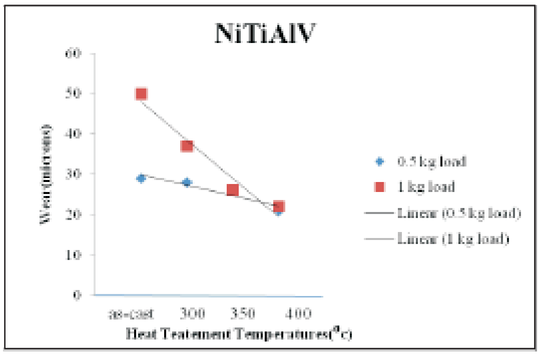


Fig (b)

Effect of heat treatment on Specific Wear Rate for all composions

The effect of heat treatment on specific wear rates for two compositions is tabulated

Table 10: Specific Wear Rate for NiTi Compositions

	Load(kg)	Speed(rpm)	Time(sec)	Specific Wear Rate($(\times 10^{-4})(\frac{mm^3}{N})$)
NiTi-as-cast	0.5	200	590	1.75
	1	200	585	1.04
NiTi-HT 300°C	0.5	200	585	1.58
	1	200	587	1.53
NiTi-HT 350°C	0.5	200	595	1.85
	1	200	590	1.60
NiTi-HT 400°C	0.5	200	545	1.76
	1	200	592	1.56

	Load(kg)	Speed(rpm)	Time(sec)	Specific Wear Rate($(\times 10^{-4})(\frac{mm^3}{N}/)$
NiTiAlV -as-cast	0.5	200	590	5.55
	1	200	585	4.87
NiTiAlV -HT 300°C	0.5	200	585	5.53
	1	200	587	3.62
NiTiAlV -HT 350°C	0.5	200	595	5.05
	1	200	590	2.54
NiTiAlV -HT 400°C	0.5	200	545	4.08
	1	200	592	2.06

The results for specific wear rates are illustrated graphically below for both compositions and heat treated conditions

Figure 16: Specific Wear Rate of NiTi Compostion

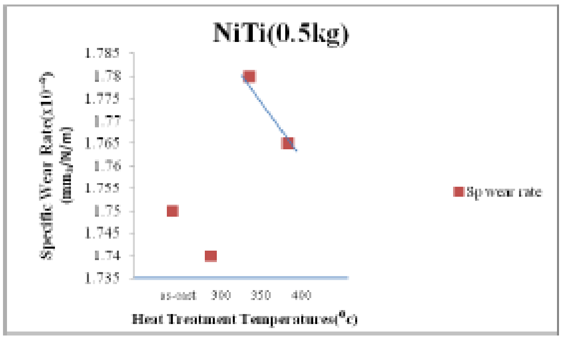


Fig (a)

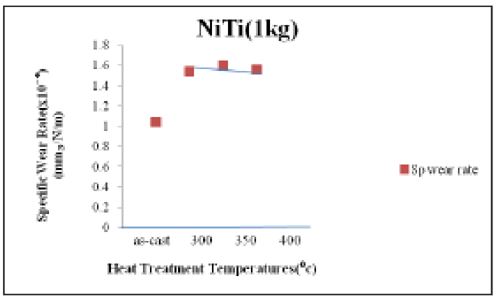


Fig (b)

CONCLUSION

The micro-structures of the specimens show fine dendrites of NiTi in the as-cast condition but grains start growing bigger and tending to homogenize as the aging temperature is increased.

Figure 17: Specific Wear Rate of NiTiAlV Composition

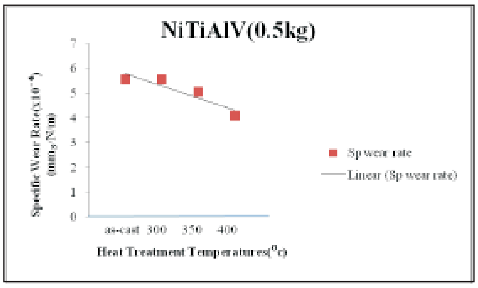


Fig (a)

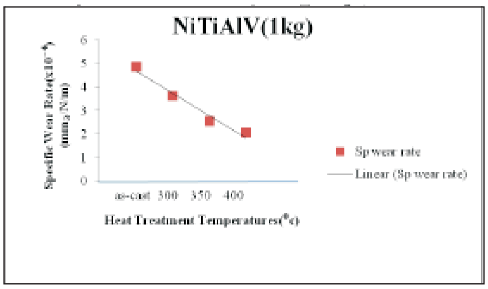


Fig (b)

The Vickers hardness is low for the as-cast samples of both the compositions but gradually increases with increase in the heat-treatment temperatures up to 350oC possibly due to formation of transition precipitates but there is a decrease in the hardness at 400oC possibly because of equilibrium precipitates NiTi formation.

Abrasive specific wear rates show a trend that is exactly the inverse of Vickers hardness. The wear rates tend to decrease from as-cast condition up to 350oC heat-treatment, and increase in the samples heat-treated at 400oC. Again possibly due to the formation of equilibrium precipitates.

The material even when heat -treated for all the three compositions has shown very marginal change i.e. increase in the at temperature since the heat treatment temperatures and times appear to be rather low.

The as-cast samples for all the three compositions are observed to have very high internal stresses in them.

The microstructures clearly show a relief of the internal stresses in the alloy samples as it is seen that the grains in the highest heat-treated condition are clearly seen compared to the as-cast ones. In the as-cast samples, the structures are seen to be in the form of dendrites that tend to change to polyhedral grains with an increase in the heat-treatment temperature to 4000 C for one hour holding.

The Vickers hardness of the samples is seen to go up gradually with an increase in the aluminum contaent.also the effect of heat -treatment will reduce the internal stresses and eliminate coring. the marginal hardness increase may be due to the Ni-Ti composition moving towards stoichiometry by precipitation of excess Ni or Ti in the form of suitable precipitates.

Abrasive wear of the specimens is seen to increase both with an increase with the cooper content and the heat-treatment temperature, for different reasons.

The specific wear rate for the samples is seen to go up accordingly with the abrasive wear since the ductility of the samples may be lowered considerably with an increased addition of aluminum to the NiTi matrix.

SCOPE FOR FUTURE WORK

The NiTi alloys contain higher aluminum concentrations need to be investigated as conflicting reports exists about the properties of NiTi alloys of higher aluminum concentration.

Effects of addition of other element s such as chromium, niobium etc to the NiTi matrix on the transformation temperature and mechanical properties can be studied.

The aging process can be investigated by varying the aging time with the aging temperature.

Influence of amounts of Ni3Ti and Ti2Ni on the aging behavior and properties of the ally need to be elucidated.

Wear test can be performed by varying other parameters such as sliding speed and also wear track diameter to study the effects on wear rate.

Micro-hardness of the alloy samples can be carried out to study the hardness at different locations at micron level.

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