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A LOOK AT NITINOL AND ITS HISTORY

DISCOVERY, APPLICATION AND PROSPECTIVE FUTURE

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Abstract

Nitinol is an alloy composed of roughly 50-50 % nickel and titanium. The alloy has significant industry application because of its properties as a shape-memory-alloy remembering its previous state from a deformed state. It is incredibly difficult to manufacture because the properties rely on the bonding on Ni-Ti and Ti tends to bond with O because of its prevalence in the Earth's atmosphere, changing the properties of the alloy.

Nitinol is extensively applied in medical applications and as an actuator in robotics fields for locomotion. Current research into Nitinol focuses on surface science and its interaction with the surface, modifying the oxide layer to be more compatible with biological interfaces. In robotics it is used as an application towards soft robots removing hard limbs.

Three potential research projects are considered by the author in medical, manufacturing and robotics fields.

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The goal of this paper is to provide a semi-exhaustive literature review of nitinol with many primary sources, provide current industry application (as of 2018) and introduce avenues that the writer (or other potential researchers) has interest in nitinol for future research as a critical thinking tool. This paper is not intended to be a primary source of nitinol but as a source aggregate.

Introduction

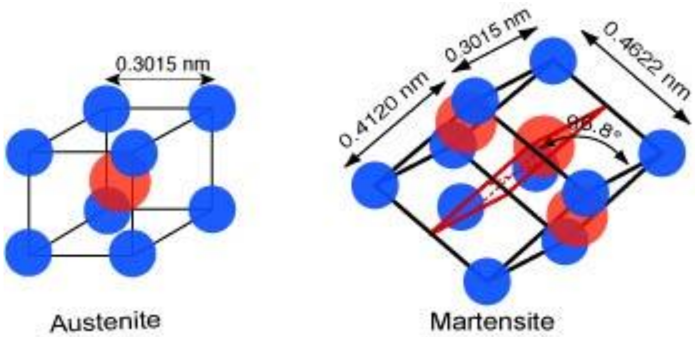

Figure 1: Cubic Structure of A-phase, B-phase

Nitinol is a metallic alloy of roughly one-to-one mixture of Titanium and Nickel metals which exhibit *shape-memory* and *super-elasticity*. Its abilities are derived from a reversible solid-state phase transformation known as a *martensitic transformation* (transformation where the unit cell of a crystal structure, usually a perfect cube, is distorted by interstitial atoms that do not have time to diffuse out during displacement transformation. Consequently, the unit cell becomes slightly longer in one dimension and shorter than the other two; a subtle change in dimension), between two different martensite crystal phases (see Figure 4: Shape Memory Process for visual). Transition requires between 10,000-20,000 psi (69-138 MPa) of mechanical stress. There is a primary *austenite (A)* and *martensitic (M)* phase at high temperature and a low temperature respectively (see Figure 1: Cubic Structure of A-phase, B-phase). A-phase is an interpenetrating simple cubic structure and is referred to as the parent phase. M-phase is a monoclinical crystal structure (Vectors of uneven lengths which form a rectangular prism with a parallelogram at its base. Two vectors are perpendicular and the other two are not). M-phase meets at 90-90-88.8-81.2 degrees. (Otsuka & Ren, 2005)

Thermal Properties

There are four distinct transition phases in two groups: A-phase-f to M-phase-s and M-phase-f to A-phase-s (see Figure 2: Transformation Temperature Relationship Char). Deformation is represented by the M-phase-s to M-phase-f and restoration by A-phase-s to A-phase-f. (Confluent Medical Technologies, 2018)

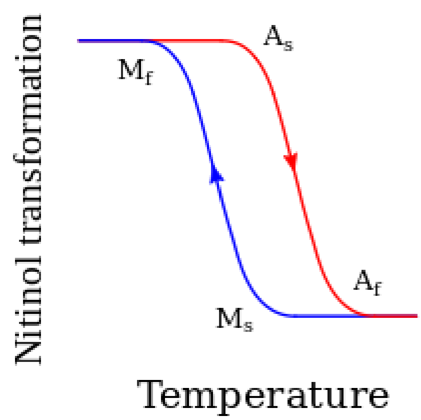

Figure 2: Transformation Temperature Relationship Chart

This cycle shows *thermal hysteresis* (dependence of the state of a system on its history-reversal of any deformation back to parent phase in context of Nitinol). The hysteresis width depends on the precise nitinol composition and processing with a typical temperature value spanning 20-50K (20-50 °C; 36-90 °F) but can be further modified by: Alloying, Processing.

Modification of Thermal Properties through Alloying, Processing

There is an ultralow-fatigue shape memory nitinol alloy (TiNiCu) with Ti_2Cu precipitates embedded in the base alloy which allow at least 10 million transformation cycles. (Chluba, et al., 2015)

Archwires analyzed	Cooling				Warming			
	Ri	Rf	Mi	Mf	Ri	Rf	Ai	Af
Nitinol Termoativado	11.60	6.55	-24.00	-42.36	---	---	13.71	24.75
NeoSentalloy F200	21.11	16.31	-24.59	-51.60	---	---	19.21	29.37
Thermo Plus	16.07	12.02	-42.72	-64.14	---	---	6.62	20.39
Copper Ni-Ti 35°C	---	---	19.34	-24.56	---	---	2.93	42.64
Flexy Thermal 35°C	---	---	35.97	16.68	---	---	10.81	45.42
Superthermal Nickel Titanium Arches	19.42	11.73	-54.73	-68.68	---	---	1.54	24.18
Heat Activated NiTi	29.68	18.56	-41.18	-64.13	---	---	8.50	33.90

Figure 3: Results from (Spini, et al., 2014) [initial austenitic (Ai), final austenitic (Af), initial martensitic (Mi), final martensitic (Mf), initial rhombohedral (Ri) and final rhombohedral (Rf)]

A study was done on seven brands of nitinol archwires (commonly used in orthodontic application on dental braces as a source of force in correcting irregularities with teeth position. (Alpern, 2018)) from seven different manufacturers and found that there was great variability in the transition temperature range and the elastic parameter of each nitinol could not be considered equivalent across manufacturers (see Figure 3). They recommend providing a datasheet for each specific product. (Spini, et al., 2014)

History

The industry workhorse standard alloy is 50.8% nickel – 49.2% titanium. (Pelton, Russell, & DiCello, 2003)

Nitinol was first synthesized in 1959 to create a missile nose cone which could resist fatigue, heat and the force of impact. In 1961, a sample was passed around in a laboratory management meeting and was passed around, noting it flexed like an accordion. One of the participants applied heat from their pipe lighter and surprised everyone when the accordion strip stretched and took its previous shape. (Withers, 2013) This demonstrated two key aspects of the alloy. First, the transformation is reversible by applying energy, and second, that it is in both directions and instantaneous. The ability for a material to be stretched and return to a previous defined state theoretically solves many problems with deformation, if the nitinol alloy can operate in requested operating conditions.

Crystal Structure and Twinning

M-phase has a monoclinical (B19) crystal structure which allows for limited deformation without breaking atomic bonds, known as *twinning* (rearrangement of atomic planes without causing sip, or permanent deformation). The one-to-one alloy can withstand about 6-8% deformation. (Funakubo, 1984)

Large amounts of pressure can be produced by preventing the material from transitioning M-phase-deformed to A-phase-parent (35,000 – 100,000 psi). This quality can be derived from the ordered intermetallic nature of the alloy. Each atom has very specific locations in the lattice structure. (Nitinol Devices & Components, 2013)

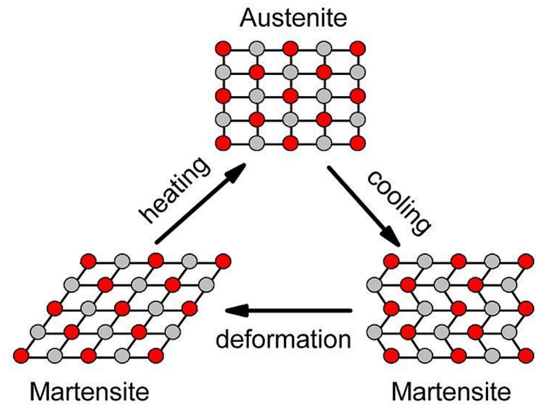

Figure 4: Shape Memory Process

Shape Setting

As seen in Figure 2 (cooling A-phase to form M-phase, deforming the M-phase to M-phase-deformed, then heating to revert to A-phase, thus returning the original, undeformed shape) is the *thermal shape memory effect*. To change the A-phase-parent, the alloy must be held in a desired position and heated to 500 °C (932 °F) called *shape setting*. (Memry Corporation, 2018)

Super Elasticity

A second effect demonstrates *super-elasticity* when M-phase is formed by either applying stress or by a cooling period. This demonstrates that within an operating temperature zone, stress can be applied to A-phase causing M-phase to form while simultaneously the environment is transforming M-phase back into A-phase because of the ambient temperature, acting as a super spring. It has an elastic range 10-30 times greater than a normal spring material such as steel. The operating temperature range is 0-40 K (0-40 °C; 0-72 °F) above A_f (between 20-50K (20-50 °C; 36-90 °F) as stated earlier in usual operating conditions with effective temperatures 20-90K depending on the specific alloy being used). Deformation above upper limit M_d (highest temperature) does not form additional M-phase-deformed and alloy experiences permanent deformation. (R Meling & Ødegaard, 1998)

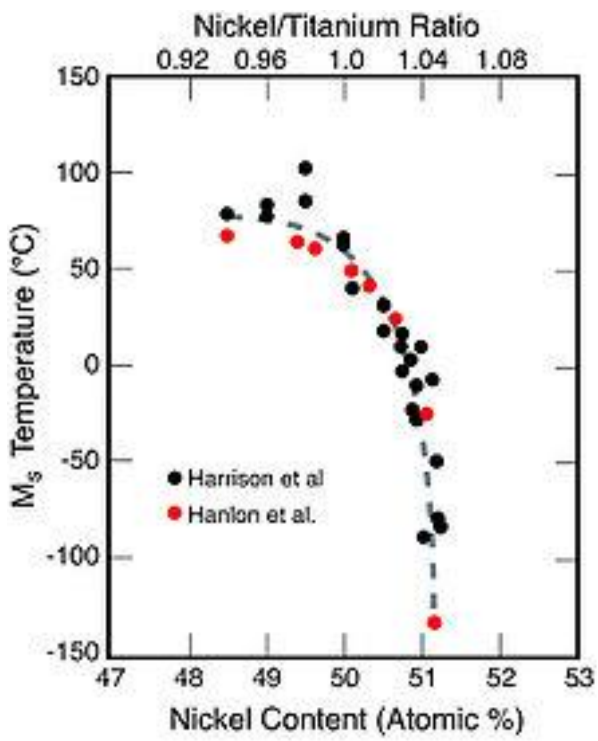

Figure 5: Composition of nitinol vs final temperature (transform from A-phase to M-phase by cooling)

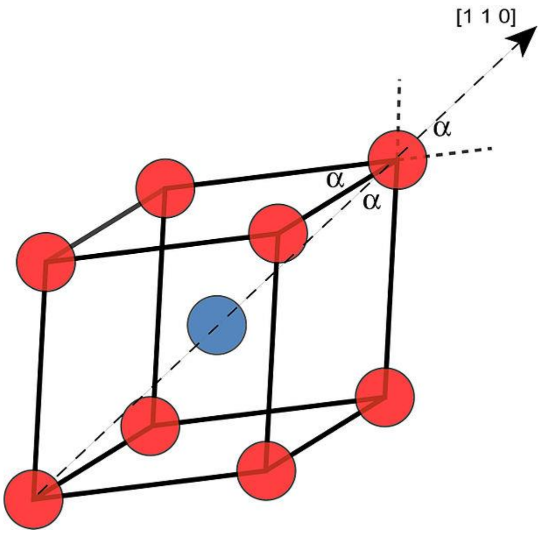

Figure 6: R-phase distortion of B2 A-phase structure

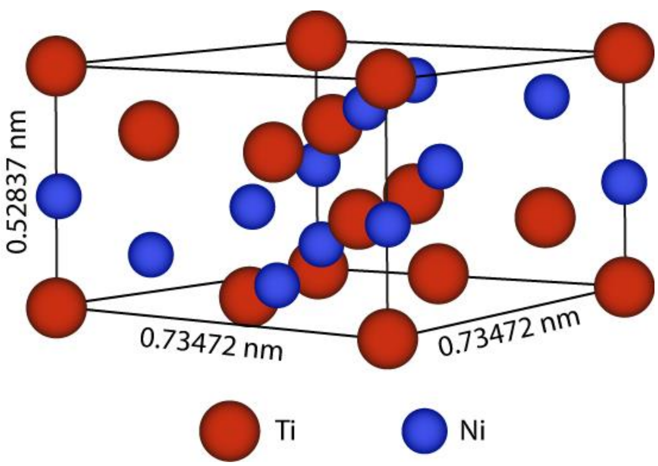

Figure 7: R-phase whole unit cell structure

Nitinol R-phase

Composition of Nitinol is approximately 50-51% Nickle by atomic percent – 55-56% percent weight. Small changes in composition can drastically change the transition temperature drastically (see Figure 5).

It is interesting to note that there is an R-phase, which is another form of M-phase but does not exhibit any significant memory effect; it is usually of nonpractical use. It is a rhombohedral distortion of A-phase (see Figure 6).

Nitinol can transform in three ways: direct transformation, symmetric R-phase transformation, asymmetric R-phase transformation.

Direct transformation shows no evidence of R-phase during forward or reverse/cooling or heating cycles and occurs in titanium-rich alloys and fully annealed conditions. Symmetric R-phase transformation occurs when R-phase is apparent in both cooling and heating cycles. In Figure 8 in Heat flow vs Temperature, two peaks are visible showing a transformation from M-phase, R-phase, and A-phase and the other way around. Note in Figure 9 shows asymmetric R-phase transformation where twin peaks are present only in cooling phase. [1]

From a well-worn phrase in device engineering and nitinol research, "It must be the R-phase" whenever a device fails to perform as expected. (T. W. Duerig, 2015)

[1] Figures from R-phase section (Figure 6 - Figure 9) are derived from https://en.wikipedia.org/wiki/R-Phase to show clarity for casual readers, however the author of these images sourced the images from (T. W. Duerig, 2015)

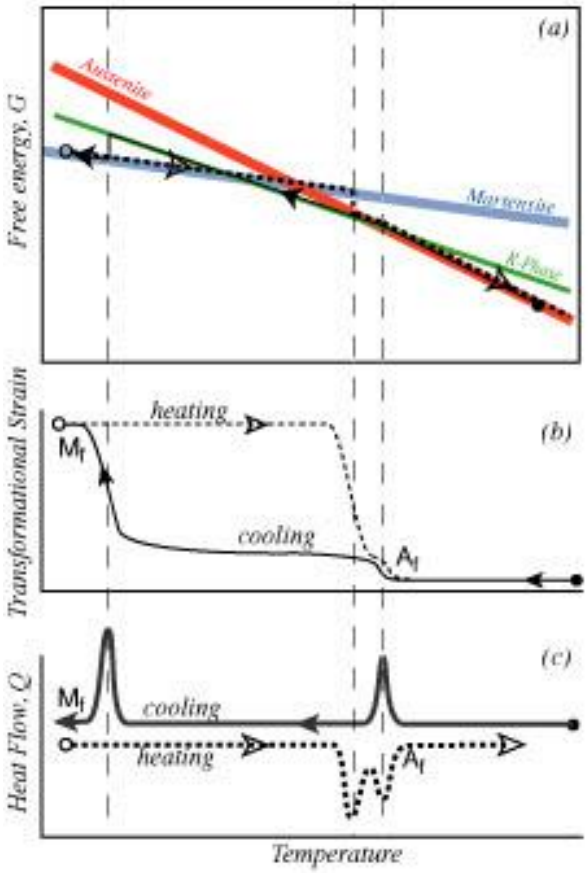

Figure 8: Free energy, strain, and calorimetry curves typical of the symmetric Austenite-R-Martensite transformation, in which R-phase is found during both cooling and heating.

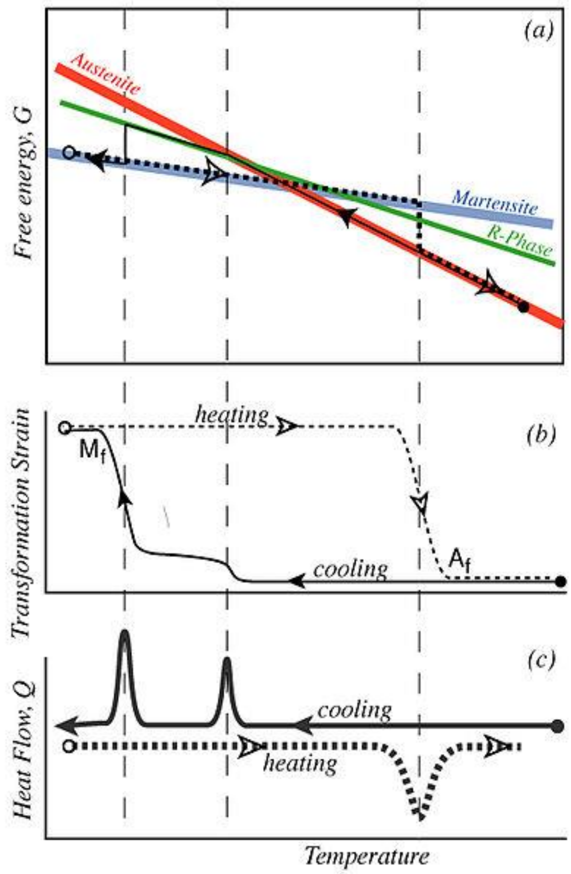

Figure 9: Free energy, strain, and calorimetry curves typical of the asymmetric Austenite-R-Martensite transformation, in which R-phase is found only upon cooling

Applications of Nitinol

Engineering application uses nitinol for demands of enormous flexibility and motion (e.g. medical devices such as heart valves or separately thermomechanical actuators) which leads to significantly greater fatigue strain when compared to other metals (such as A36 structural steel). There is limited understanding of fatigue failure for the absolute limits of nitinol.

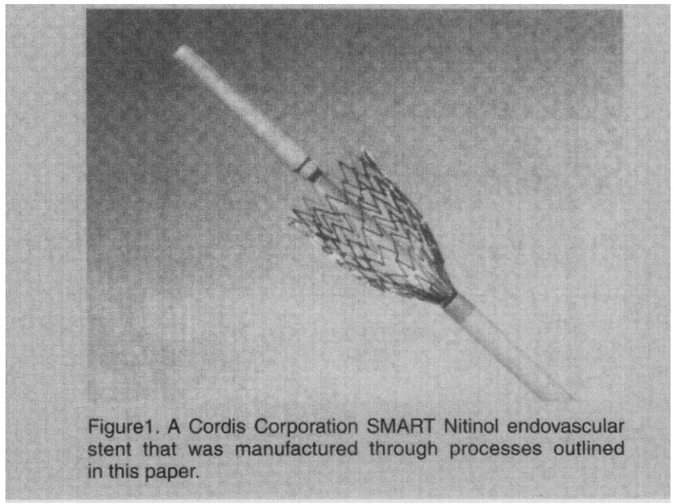

Figure 10: Example of medical application, sourced from (Pelton, Russell, & DiCello, 2003)

Medical Application from Titanium Oxide and Alloy Issues from Nickel

Nitinol inherits the metallic properties of their respective components, and nickel is known to be an allergen and a carcinogen (due to its wide use in medical application (Pelton, Russell, & DiCello, 2003)). It can be treated via electropolishing (removes material from a metallic workpiece to smooth the surface) or passivation (becoming less affected or corroded by the environment of future use), thus creating a stable protective layer of TiO_2 which acts as a self-healing barrier against ion exchange. (see Figure 12) (Clarke B.1, 2006)

Table II. Oxide Thickness Values (in nm) and Outer Surface Chemical Composition as Determined by Auger Spectroscopy (Atomic %) for Wire Samples

Sample No.	Oxide Thickness (nm)	Outer Surface Chemical Composition (Atomic %)								
		C	O	Ti	Ni	Ca	Cl	K	Na	S
1	340	33.9	33.9	7.3	15.2	n/d	4.5	n/d	2.9	2.3
2	120	41.6	38.8	12.0	3.6	n/d	0.7	0.6	1.6	1.0
3	170	25.2	52.1	17.3	1.8	0.7	0.2	0.5	1.4	0.8
4	15	24.7	51.6	15.7	1.3	3.3	0.2	0.8	1.9	0.4
5	11	19.2	57.5	16.0	n/d	2.5	0.1	1.3	3.3	0.3
6	14.5	22.9	60.1	11.3	1.7	1.2	0.1	n/d	2.4	0.3
7	16	26.5	52.0	17.9	1.1	1.1	0.2	1.1	n/d	0.2

Figure 11: Samples derived from (Clarke B.1, 2006) for context for Figure 12

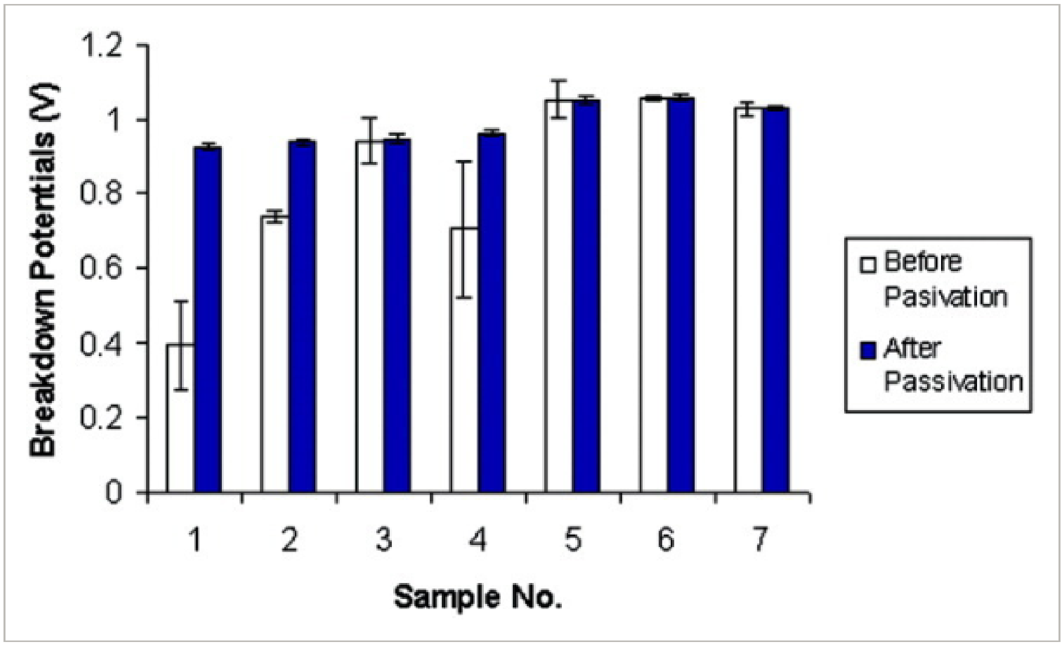

Figure 7 Open in figure viewer | PowerPoint

Effect of passivation on breakdown potentials. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com]

Figure 12: "It is clear that for the samples with low initial pitting potentials, considerable improvement in corrosion resistance is observed. For the more stable samples, little change was observed." (Clarke B.1, 2006) Note samples 5 – 7, with O context above 50%

It is suggested that the size, distribution and type of inclusions (chemical compounds and nonmetals that are that are present in alloys, such as the oxide layer with titanium) can be controlled to some extent. Theoretically, smaller, rounder and fewer inclusions should lead to increased fatigue durability and current studies suggest a relationship between fatigue resistance and typical inclusion size in an alloy. (Urbano, Coda, Beretta, Cadelli, & Sczerzenie, 2013)

Inherited Issues from Titanium

Nitinol is difficult to weld because it inherits properties of titanium. Titanium and titanium-based alloys are plagued with poor quality and highly brittle welds due to the formation of Ti-Fe intermetallic in the weld pools. This has been mitigated by using fillers, such as nickel or ion in the pool and different welding techniques such as laser welding. (USA Patent No. US2004182835 (A1), 2004)

Current Research

The properties of nitinol lend itself to be used as an actuator, as seen in its extensive medical applications. The frequency of actuation is dependent on the heat management, especially during cooling phase (see Figure 8, Figure 9 for visual). There are numerous methods used to increase the cooling performance, such as forced air[2], flowing liquids[3], thermoelectric modules[4], heat sinks[5], conductive materials[6] and higher surface-to-volume ratio[7]. The current fastest nitinol actuation is a few milliseconds from a complete phase transformation by a high voltage capacitor discharge which rapidly heated the material (as a smart memory allow wire). (Vollach, 2010)

Table 3. Heat transfer coefficients for various active cooling methods for Flexinol wire.

Cooling method	Constant heat transfer coefficient $h = h_0$ (W/m^{2}K)	Variable heat transfer coefficient ($h = h_0 + \beta T^2$)
Fluid quenching	2800	–0.3
High speed conv.	800	–0.1
Heat sink	650	–0.09
Low speed conv.	300	–0.04
Free	185	–0.02

Figure 14: Example for forced air cooling of Flexinol wire from (Tadesse Y, 2010), see graph at Figure 19

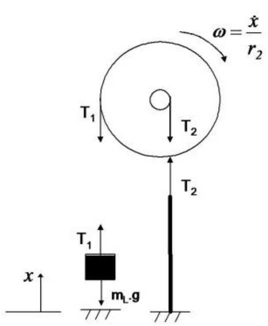

Figure 16: Mechanical mechanism to measure A by modifying x and inducing stress in the SMA wire from (Romano R, 2009)

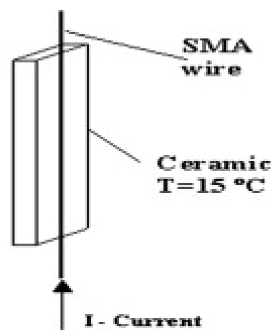

Figure 13: SMA wire and cooling element from (Romano R, 2009)

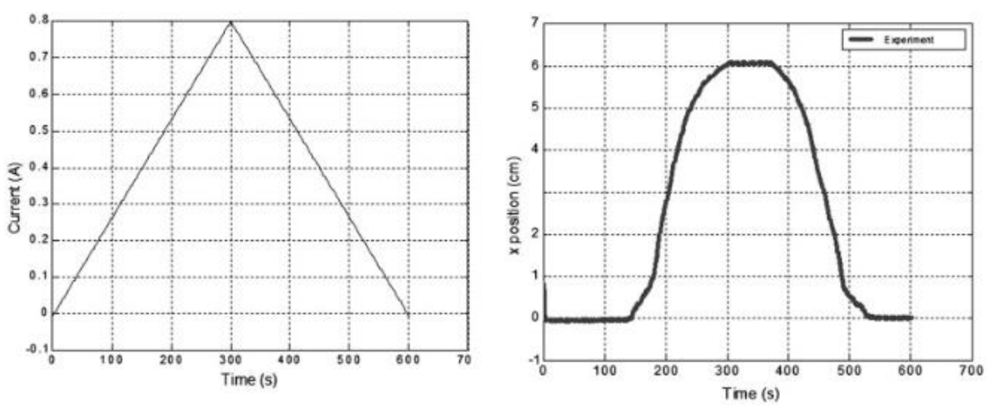

Figure 15: Relationship between A and x demonstrated from Figure 16

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[2] (Tadesse Y, 2010)
[3] (Wellman PS, 1997)
[4] (Romano R, 2009)
[5] (Russell RA, 1995)
[6] (Chee Siong L, 2003)
[7] (An L, 2008)

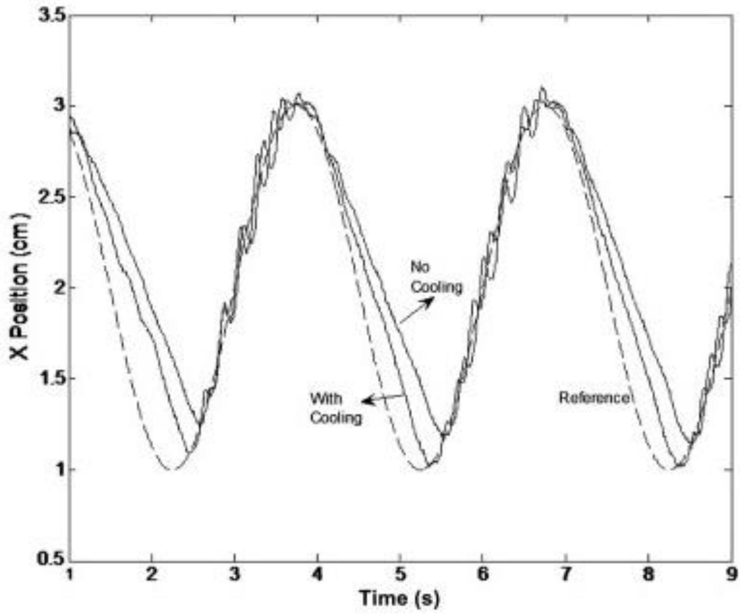

Figure 18: Harmonic set-point response of the SMA actuator (1 cm amplitude), 3 s period, with and without cooling. (Romano R, 2009) showing faster response with active cooling

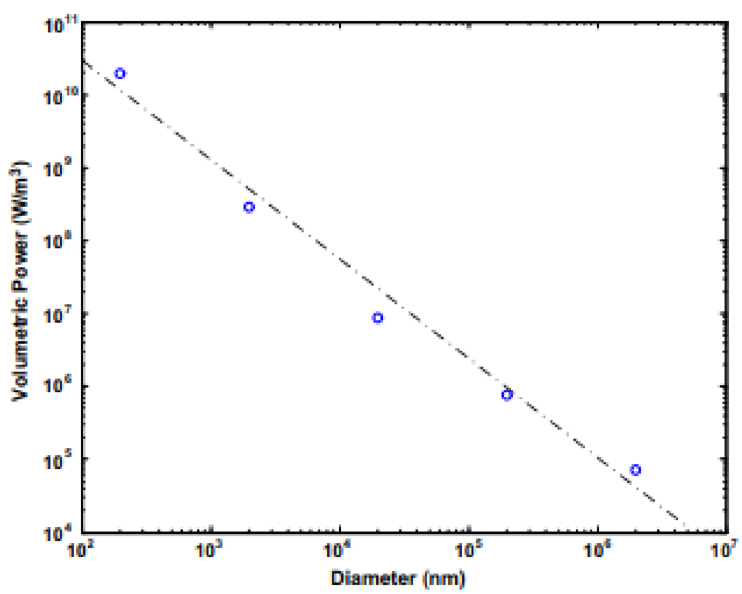

Fig. 12. Diameter (d) vs. volumetric average power (symbol: simulation; dash-doted line: data-fitting).

Figure 17: Diameter (d) vs. volumetric average power (symbol: simulation; dash-dotted line: data-fitting) (An L, 2008). Note, larger diameter, larger surface area, lower power density

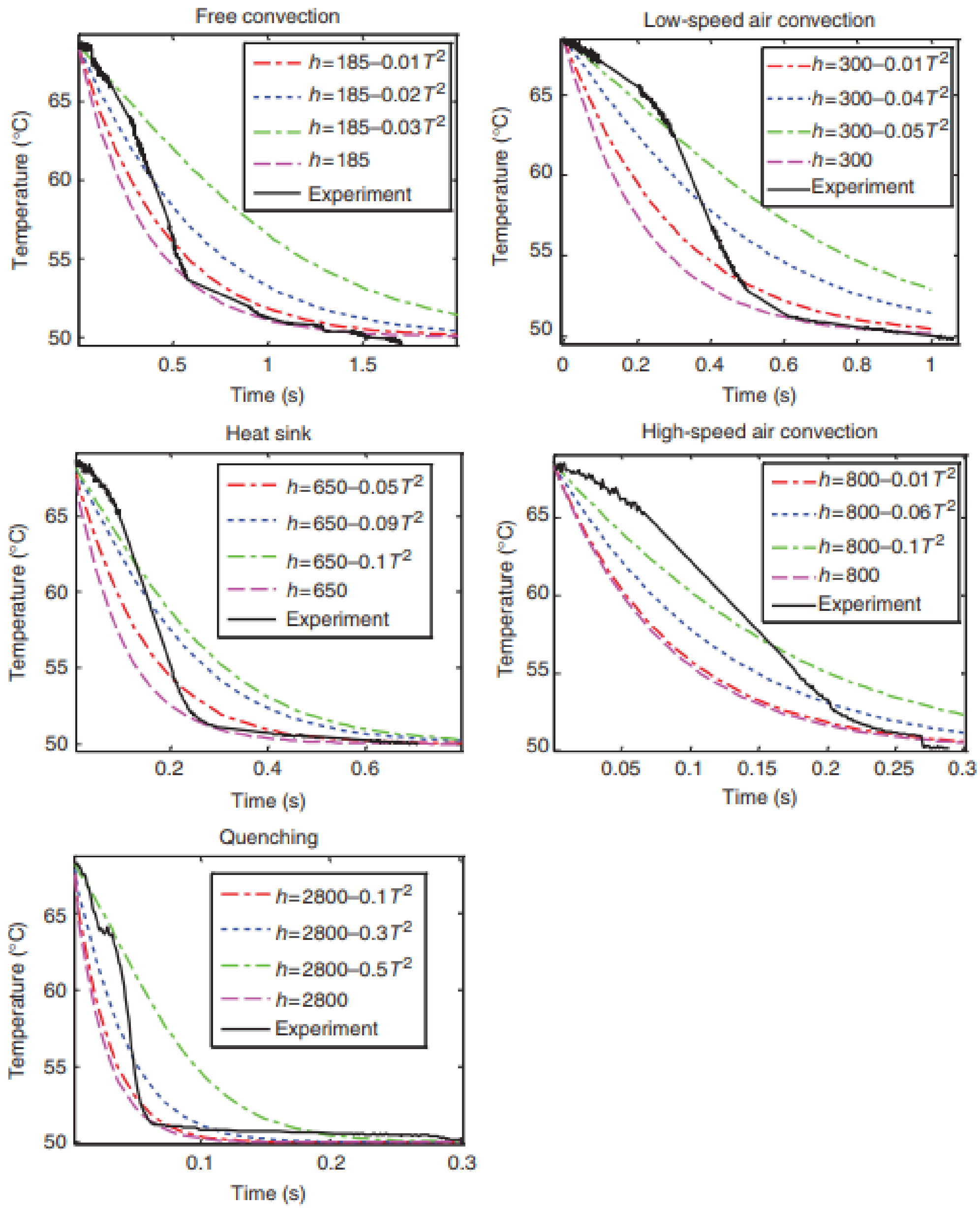

Figure 15. *Constant and variable convective heat transfer coefficients for various active cooling techniques.*

Figure 19: Graphs showing heat transfer coefficient as a function of temperature vs time for five cooling methods from (Tadesse Y, 2010)

Current Industry Adoption Issues

Interestingly, even though from the conception of the alloy showed immediate application, industry adoption has been slow due to the various difficulties that existed in manufacturing this alloy. Each atom of titanium that is in contact with the atmosphere creates the oxide layer which protects against corrosion, but has an inverse relationship with the *shape-memory* properties of the alloy, not bonding in the crystal structure as seen in Figure 1. Looking at Figure 11,

"All nitinol samples used in this study were prepared by Fort Wayne Metals Research Products Corporation, Indiana, specifically for this study. Nitinol wire samples with a diameter of 0.762 mm were fabricated from binary nickel–titanium alloy with a nominal composition of 50.8 atomic percent nickel and an austenite start temperature in the fully annealed condition of −31°C as measured by DSC per ASTM F 2004–00. Standard reduction and thermal processing was used to draw the wire to 1.02 mm. Additional processing to achieve 45% cold work at 0.762 mm followed by a heat straightening step to produce super elastic properties at room to body temperature were performed. " (Clarke B.1, 2006)

Despite being manufactured at the same time, in the same batch, with the same methodology, there is enough difference between each sample to show noticeable results as seen in (Clarke B.1, 2006). Issues like this study emphasize using extreme high-purity raw materials to synthesize the alloy specific to a case-scenario, such as in Figure 5. This creates a state where there is an industry standard (50.8% nickel – 49.2% titanium, cited from earlier in the paper) and there exists custom alloys, leading to financial restriction as one of the few barriers to using a *shape-memory-alloy* in all cases.

Writer's Critical Thinking & Possible Research Topics

Nitinol has been used to address an issue in robotics and hard limbs by considering artificial muscles[8] [9], a 'soft robot' worm[10], control of industrial robotics by replacing limbs with nitinol rods[11], underactuated robot finger[12], a method to fabricate *shape-memory-alloys* on elastomeric polymer substrates to function as actuators for soft body robots[13] and a proof of concept for inexpensive locomotion by a simple hexapod[14].

An intended area of study is mechatronics and material science, utilizing new materials and taking advantage of computer science to accelerate human progress.

[8] (Kim, 2009)
[9] (Tondu, 2007)
[10] (ANDERSEN, 2017)
[11] (EHRAT, 2017)
[12] (Dessauw, 2013)
[13] (P.D. Fallon)
[14] (Mills)

Results and Discussion

This paper will generally focus in the field of applied robotics and application of nitinol as a potential application area. Some primary sources have been referenced in “Writer’s Critical Thinking & Possible Research Topics”.

Proposed Research Topic: Application of Nitinol Muscles and Neural Networks to copy Human Motion of Grabbing Objects

In the interest of the writer, nitinol can be utilized as an actuator simply by applying an electric current to induce resistance in the SMA wire, or as a sensor by measuring the current produced by changing the tension in a SMA wire, as shown by (Romano R, 2009). This has extensive application in robotics. Currently, there is a still a distance between robotic movement and the concept of automating humanity. Robots are inherently rigid, with traditional robotics utilizing stepper, servo, and brushless motors to move limbs without an exponentially increasing degree of freedom due to an increasingly infinite number of joints to be controlled by limited onboard embedded computers.

If the writer were to consider a research topic involving Nitinol, it would involve the further application of nitinol actuators in robotics utilizing neural networks[15] to learn to grab objects like humans through a two-phase concurrent research study: creating a neural network (most likely a Deep Convolutional neural Network to process visual data and then transition to another form to encourage self iteration) to ‘design’ an actuating arm based on the actions of humans using their appendages to hold objects. Another neural network (Unsure which network would be best suited and this requires additional research that is beyond the scope of this paper) most likely to iterate on the design to optimize the task assuming the ‘muscles’ are nitinol rods of varying diameter and length. This process deeply relies on the property discussed at Figure 5, by varying the ratios of Nickle and Titanium and considering any inclusions to tweak the properties of nitinol muscle.

This may most likely create complex assemblies of nitinol, most likely of varying diameter, thickness, and changing composition and a similar elastomeric polymer to mimic bone structure that is both rigid and flexible enough as a framework to artificially hold objects like humans.

The assembly would be further optimized by generative design, a relatively new form of computer aided design which considers materials, manufacturing methods, cost constraints and loads.

The second stage would be automating the previous processes into a single chain that can be further optimized by neural net optimization.

After self iterating for a new thousand generations, modern manufacturing methods such as 3D resin printing and computer controlled molten metal mixer (unsure if this even exists) can create these complex parts and the assembly can be created from proof of concept to physical proof.

Proposed Research Topic: Refinement of Nitinol Manufacturing

However, the proposed study above is close to impossible if specific alloys of nitinol are required and cannot accurately manufactured rapidly and in bulk quantity to allow for economies of scale to positively affect the world in practical research. Research in this case would involve creating perfect laboratory conditions to manufacture nitinol to a greater exact percentage, such as down to each individual atom.

[15] Use this webpage as a reference for common neural networks: https://towardsdatascience.com/the-mostly-complete-chart-of-neural-networks-explained-3fb6f2367464

This would require easy manipulation of metals at the atomic scale and point to quantum mechanics for inspiration.

Current manufacturing methods take advantage of VIM (Vacuum Induction Melting: uses alternating magnetic fields to heat the raw materials in a crucible in a very high vacuum) and have demonstrated smaller inclusions than VAR (Vacuum Arc Remelting: uses electrical arcs between raw material and a water-cooled copper strike place taken place in a high vacuum into a water-cooled copper mold), showing that VIM manufacturing methods currently give a higher fatigue resistance. (Urbano, Coda, Beretta, Cadelli, & Sczerzenie, 2013)

A research proposal on this concept would be to identify ways to directly manipulate bonds between atoms and to cherry pick and create the crystal structure atom by atom.

Proposed Research Topic: Nitinol Biocompatibility

An interesting aspect of Nitinol is not too knowledgeable about is medial applications of material science. Due to the creation of Titanium oxide on the surface, nitinol has exceptional biocompatibility because it is corrosion resistant. This has led to research in implanted devices that can remain in the body for indefinite periods of time such as a heart stent, which remains in the blood vessel for a year or more in additional to an antiaggregant drug therapy, to prevent the formation of a clot around the stent. After drug therapy, the stent becomes integrated into the blood vessel wall.

Much of the research done with nitinol has been on surface science and the interaction of the coating to its environment. Research is currently underway to produce coatings that are as close to biological material as possible to minimize the foreign body reaction that can occur in implants. (Brassack, 2000)

Brassack explores the use of silica and various bone-relevant proteins such as collagen, gelatin and commercial collagen hydrolysate to coat metallic alloys with implanted applications.

Research in this field would require a strong understand of human chemistry and the reaction rates to various materials as an average in populations to certain kinds of coatings. It may require some proteins to be region specific and/or catered to the individual, creating the coating from the patient's own biological material to minimize foreign body risk.

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