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INTERMETALLIC MICROSTRUCTURAL AND MECHANICAL RESPONSE OF NITI (NITINOL) SHAPE MEMORY ALLOY WITH LASER WELDING TO VARIOUS MATERIALS

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***Abstract*: In this ever-changing and evolving world, the demand for material goods is ever increasing. Certain metals and materials have become highly sought-after. The demand for new alloys for various commercial and medical applications increases every day [1]. To address the new age industry demands, focusing on longevity, versatility, extreme load case scenarios, and commercial viability, scientists have to develop new manufacturing techniques and methods. This will enable them to create and mass-produce various new metal alloys that can meet the demands for the designs of new products and replace traditional elements, such as carbon steel or aluminum, in existing products with an ever-increasing workload.**

This paper will focus on the discovery, development, evolution, manufacturing, uses, and commercial viability of Nitinol (NiTi) and what impact it has had due to its unique properties across many industries.

Super-elastic Nitinol has become a familiar and well-known engineering material in the medical industry over the last couple of decades. While the greater flexibility of the alloy drives many of the applications, there are many lesser-known advantages of Nitinol in medical devices. [1, 2, 3]

***Keywords:* Nitinol, Properties, Welding, Shape Memory Alloy, Mechanical Response, Medical Devices**

I. INTRODUCTION

Nitinol (NiTi) was initially developed for military purposes by funded research. Looking for metals, mainly metal alloys that could meet specific criteria for heat resistance, metal fatigue, and relative rigidity, this alloy was developed at the Naval Ordnance Laboratory in Maryland, the USA, by William Buehler and Frederick Wang in 1959. Having found that nickel and titanium could meet the goals they had set out to achieve, further experiments led them to conclude that a 1:1 alloy of Nickel and Titanium met the goals they had set out to achieve. Further experimentation and understanding of the alloy took another two years. Finally, a test ready sample of the alloy was presented in a meeting where the alloy showed its remarkable properties that would later make it vital for the development of several industries and a whole range of products that would not exist without it.

While the alloy's unique properties and potential applications were immediately apparent, it took more than a decade for its commercial adoption. The biggest challenge in the commercialization of this alloy was the incredible difficulty of its formation process. [1]

Mainly, the metal's melting, processing, and machining were some of the challenges in its development. In addition, due to the significant reactivity of titanium, it took a much longer process to devise a method that could be reliably used to form the metal and make it commercially viable, as earlier attempts at the same had not seen much success. As a result, it was well into the 1980s before the processes for making Nitinol became commercially successful, which saw an increase in application and formation of entirely new product lines based on this unique alloy.

The shape memory effect is not unique to Nitinol specifically, as it was initially observed in Copper-gold

alloys as early as the 1930s. However, the consistency and accuracy of the shape memory property have been the most consistent with this alloy across multiple uses and applications.
Nitinol has super-elasticity properties due to the different crystalline structures of its base metals. The super-elasticity of this metal is 10 to 30 times more than any other ordinary metal, again making Nitinol very unique.

Properties
Nitinol exhibits properties unique to itself only, while it has a few other properties that it shares with other metals. However, Nitinol excels at these, usually by a more significant margin.
Physically, Nitinol has a bright silver appearance. It has an ultimate tensile strength between 754 and 960 MPa. A unique physical property of Nitinol is its typical elongation to fracture, which is 15.5%. For comparison, steel has an extension to fracture of 0.5%.
At room temperature, Nitinol has an ultimate tensile strength of between 103 and 1,100 MPa. In comparison, steel possesses a tensile strength of anywhere between 300 and 2,400 MPa, depending on the particular steel type and composition. Above its transformational temperature range, Nitinol has been known to become significantly stronger, regardless of its various alloys. However, it's not the strength but rather the formability property of the 1:1 Nitinol alloy that makes it extremely useful for medical equipment makers. [1, 3]

For medical equipment makers, primarily a super-elastic nitinol wire rope manufacturers, the three most coveted features of Nitinol are its:

- Excellent thermal conductivity
- Biocompatibility, meaning it can be implanted within the human body without placing the patient at risk
- High corrosion resistance (more so than stainless steel)

Nitinol can be stretched very easily and deformed to serve as useful in cable manufacturing on its own. Usually, nitinol cablesare composed of a central strand of Nitinol wrapped in six non-shape memory alloys such as stainless steel [4, 5, 6]. The composition of conventional wires adds strength to the cable's construction and helps with cost reduction/effectiveness. At the same time, it makes the expensive Nitinol durable, meaning equipment containing nitinol cable lives longer and is made stronger over more cycles, thus resulting in the product with Nitinol having a much longer useable life cycle duration. [2]

Some of the significant physical, electrical & mechanical properties of Nitinol are given in the below tables.

Nitinol Physical Properties

Melting point		**1300 deg. C (2370 deg. F)**
Density		6.45 g/cu.cm (0.233 lb/cu.in)
Thermal conductivity	austenite	0.18 W/cm * deg. C (10.4 BTU/ft * hr * deg. F)
	martensite	0.086 W/cm * deg. C (5.0 BTU/ft * hr * deg. F)
Coefficient of thermal expansion	austenite	11.0E-6/deg. C (6.11E-6/deg. F)
	martensite	6.6E-6/deg. C (3.67E-6/deg. F)
Specific heat		0.20 cal/g * deg. C (0.20 BTU/lb * deg. F)
Corrosion performance		Excellent[7]

Electrical And Magnetic Properties

Resistivity [resistance = resistivity * length / cross-sectional area]	austenite	approx. 100 micro-ohms * cm (39 micro-ohms * in)
	Martensite	approx. 80 micro-ohms * cm (32 micro-ohms * in)
Magnetic permeability		< 1.002
Magnetic susceptibility		3.0E6 emu/g

Nitinol Transformation Properties

Transformation temperature		-200 to 110 deg. C
Latent heat of transformation		5.78 cal/g
Transformation strain (for polycrystalline material)	for a single cycle	max 8%
	for 100 cycles	6%
	for 100,000 cycles	4%
Hysteresis		30 to 50 deg. C

Nitinol Mechanical Properties

Young's modulus****	**Austenite**	**approx. 83 GPa (12E6 psi)**
	Martensite	approx. 28 to 41 GPa (4E6 to 6E6 psi)
Yield strength	Austenite	195 to 690 MPa (28 to 100 ksi)
	Martensite	70 to 140 MPa (10 to 20 ksi)
Ultimate tensile strength	fully annealed	895 MPa (130 ksi)
	work hardened	1900 MPa (275 ksi)
	Poisson's ratio	0.33
Elongation at failure	fully annealed	25 to 50%
	work hardened	5 to 10%
Hot workability		quite good
Cold workability		challenging due to rapid work hardening
Machinability[8]		complicated, abrasive techniques preferred

Experimental Procedure

1. Laser welding Processes

Laser micro-welding (LMW) is extensively used in manufacturing because of the advantages of welding complex geometries and unique materials. However, LMW of Nitinol is considered difficult because its functional properties (super-elasticity and shape memory effect) are sensitive to change due to the thermal impacts and chemical contamination which laser welding can induce [6]. There are limited studies detailing the mechanical properties of neodymium-doped yttrium aluminium garnet (Nd: YAG) laser-welded Nitinol. In general, laser-welded Nitinol exhibited good retention of base material strength. [9]
However, different results were reported regarding the super-elastic properties and fracture mechanisms. For example, Schlossmacher investigated Nd: YAG laser welding of Nitinol sheets, observed no super-elasticity deterioration and found that fracture occurred ductile. In contrast, a study on Nd:YAG butt welded Nitinol wires reported lower critical stress for stress-induced martensite (SIM) transformation and an increased residual strain upon unloading while observing a brittle fracture in the weld [9]. Similarly, a study by Ogata et al. reported that a brittle fracture mode occurred [10].The functional properties (super-elasticity and shape memory effect) of Nitinol are strongly linked to its ability to transform between martensite and austenite. An earlier experiment observed a slight decrease in transformation temperature after CO2 laser welding of Nitinol. [11, 12]

Preparation

The manufacturing of Nitinol presents several unique challenges because:

- Extreme precision and cleanliness are required for and during the melting process
- The standard techniques for hot and cold working Nitinol are known to be different
- The traditional product forms available
- Methods for shaping Nitinol parts
- Strategies for overcoming the difficulties of machining Nitinol parts
- Proven techniques for joining Nitinol components
- Several ways to coat or finish the surfaces of Nitinol devices

Various kinds of Methods

Nitinol is exceedingly difficult to make due to the exceptionally tight compositional control required and the tremendous reactivity of titanium. In addition, every atom of titanium that combines with oxygen or carbon is an atom that is robbed from the NiTi lattice, thus shifting the composition and making the transformation temperature that much lower. As a result, there are two primary melting methods used today:

1. Vacuum Arc Remelting (VAR)

This is done by striking an electrical arc between the raw material and a water-cooled copper strike plate. Melting is done in a high vacuum, and the mould itself is water-cooled copper.

2. Vacuum induction melting (VIM)

This is done using alternating magnetic fields to heat the raw materials in a crucible (generally carbon). This is also done in a high vacuum.

While both methods have advantages, it has been demonstrated that an industrial state-of-the-art VIM melted material has smaller inclusions than an industrial state-of-the-art VAR one, leading to higher fatigue resistance. Other researchers report that VAR employing extreme high-purity raw materials may lead to reduced inclusions and, thus, improved fatigue behavior. Other methods are also used on a boutique scale, including plasma arc melting, induction skull melting, and e-beam melting. Physical vapour deposition is also used on a laboratory scale.
Hot-working of Nitinol is relatively easy, but cold working is difficult because the enormous elasticity of the alloy increases the die or roll contact, leading to tremendous frictional resistance and tool wear. For similar reasons,

machining is challenging—to make things worse, the thermal conductivity of Nitinol is poor, so heat is difficult to remove. Grinding (abrasive cutting), Electrical discharge machining (EDM), Electrochemical machining and laser cutting are relatively straightforward.

Heat treating nitinol is delicate and critical. It is a knowledge-intensive process to fine-tune the transformation temperatures. Ageing time and temperature control the precipitation of various Ni-rich phases, thus controlling how much nickel resides in the NiTi lattice; ageing increases the transformation temperature by depleting the nickel matrix. Therefore, the combination of heat treatment and cold working is essential in controlling the properties of nitinol products.

Regardless of the fundamental difference between the two melting processes, wires manufactured from VAR and VIM/VAR double melt appear to have similar mechanical and fatigue properties.

Mechanical Testing

Nitinol has been difficult to work with and causes significant tool wear. Still, conventional methods can be applied when machining Nitinol like milling, turning and drilling. Shearing and blanking are pretty effective with proper tool design and maintenance. Carbide tools with a chlorinated lubricant are recommended for these operations. Abrasive processes such as grinding, sawing and water jet cutting with abrasive particles are successfully used for Nitinol. For example, tips of Nitinol guide wires are commonly tapered by centerless grinding. Laser machining, electro-discharge machining (EDM), and photochemical etching processes are used to fabricate various components from Nitinol. [13, 14] Stents, baskets and filters are some of the examples.

Laser machining has become the standard process for manufacturing Nitinol tubular stents. Modern laser cutting machines equipped with a CNC motion control system offer high speed, high accuracy and the capability for rapid prototyping.

However, some of the significant drawbacks of this technique are the occurrences of the heat-affected zone (HAZ) and micro cracks. Managing heat and shape-stability of Nitinol parts is critical, and post-processing is required to remove issues like slag, micro cracks and HAZ. Electro-discharge machining works well with most Nitinol compositions.[15]

Super-elasticity and shape memory effects are generally well preserved in these welding processes. Ultrasonic solder joint of Nitinol using Sn-based solder has also experimented with good results. Joining Nitinol to dissimilar metals is significantly more challenging. For example, welding Nitinol to stainless steel [4, 5] is complicated due to the formation of brittle intermetallic compounds. These oxide layers can be removed by mechanical means such as grit blasting and polishing. By proper selection of polishing media, a mirror-like finish can be achieved by mechanical polishing. Chemically etching, also effective in removing surface oxide, produces a silver-looking surface. Electro-polishing of Nitinol has also been demonstrated to create a highly smooth finish. The corrosion resistance of Nitinol is significantly affected by methods of surface preparation. It is generally believed that the preferential formation of titanium oxide on the surface enhances passivity and corrosion resistance. [12]

3. Weld Interface

Microstructural Analysis

The two most unique properties of Nitinol, namely Shape Memory Effect (SEM) and Super-Elasticity Nitinol, can be attributed to its unique alloy composition and microstructure formation.

The unique factor with this particular metal is its consistency of performance, which can be attributed to the consistency and stability of its microstructure, which gives it two different phases or states of existence.

Nitinol's unique physical properties are derived from a reversible solid-state phase transformation known as a martensitic transformation. The metal can transform between two different martensite crystal phases, requiring 10,000–20,000 psi (69–138 MPa) of mechanical force or stress.

When subjected to higher temperatures, Nitinol forms an interpenetrating simple cubical structure referred to as austenite, also known as the parent phase. However, at low temperatures, Nitinol spontaneously transforms into a more complicated monoclinic crystal structure known as martensite or daughter phase. It has been determined that there are four transition temperatures associated with the austenite-to-martensite and martensite-to-austenite transformations in Nitinol. [16]

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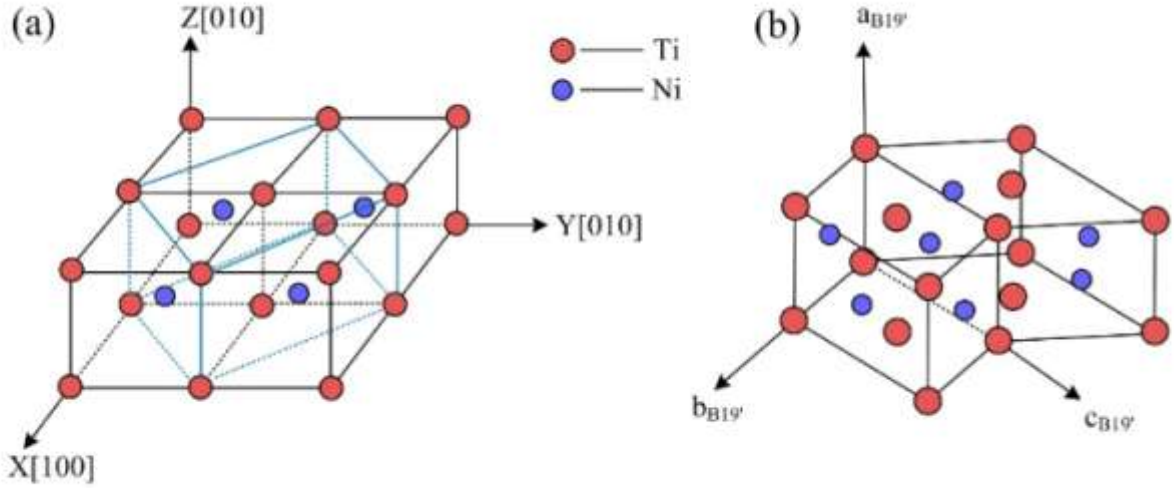

Figure 1: Crystal structure phases of NiTi showing (a) B19′ martensite and (b) B2 austenite.

SEM Microstructure

Nitinol Welding

While Nitinol offers numerous benefits, it can cause big headaches for engineers. For instance, inadequate processing can lead to premature failure and poor fatigue [17]. The material is also sensitive to heat treatments and can be difficult to join to stainless steel and other metals. [4, 5]

A variety of joining methods can be used to assemble nitinol parts. However, each process has pros and cons that engineers must consider. [18]

Properties of the Weld–Crystalline Structure

Nitinol is known to be a rigid material. It has a rugged, stable oxide layer and a high alloying content of titanium. The hardness and oxide layer causes various challenges with solid-state joining processes.

The oxide can be part of the melt pool in a laser beam. The alloy's titanium component makes it metallurgically challenging to fuse weld to non-titanium alloys with Nitinol. Titanium is known to form brittle intermetallic compounds when joined via a fusion process to dissimilar materials.

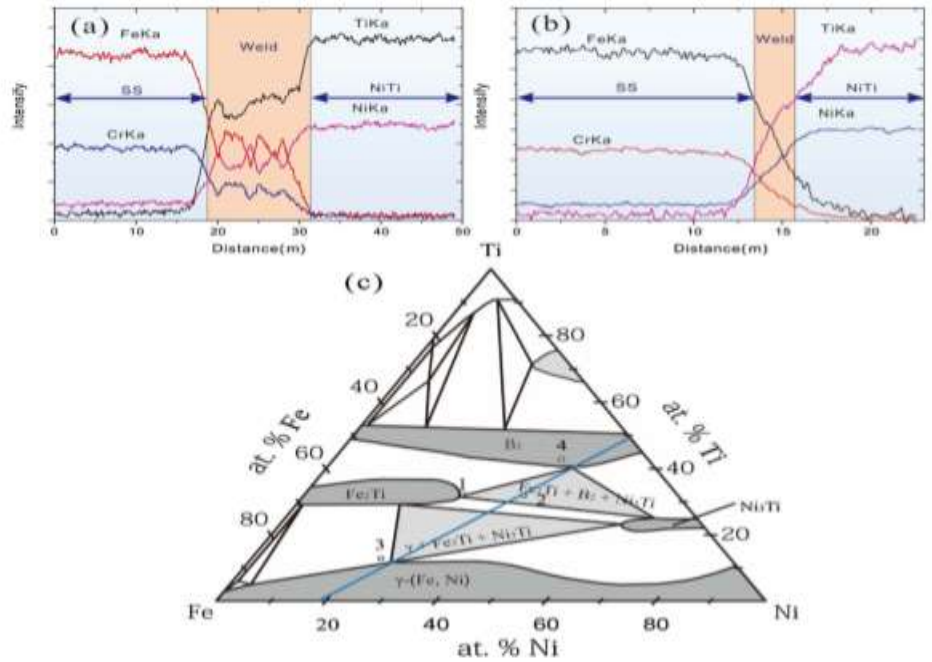

Figure 2: The blue line in the isothermal section connects the two base metals, and indicates the phases that are potentially present in NiTi/SS joints.

Solid-state resistance welding is ideal when working with an overlap joint to weld Nitinol to itself or other metal combinations. Laser welding, a fusion-based process, can effectively join Nitinol to itself and with other precious metals, such as platinum.[13]

Welding nitinol to stainless steel is considerably more difficult because of the brittle inter-metallics that form at the weld zone. In addition, it usually requires a consumable filler material to be able to create any durable weld between the two metals.

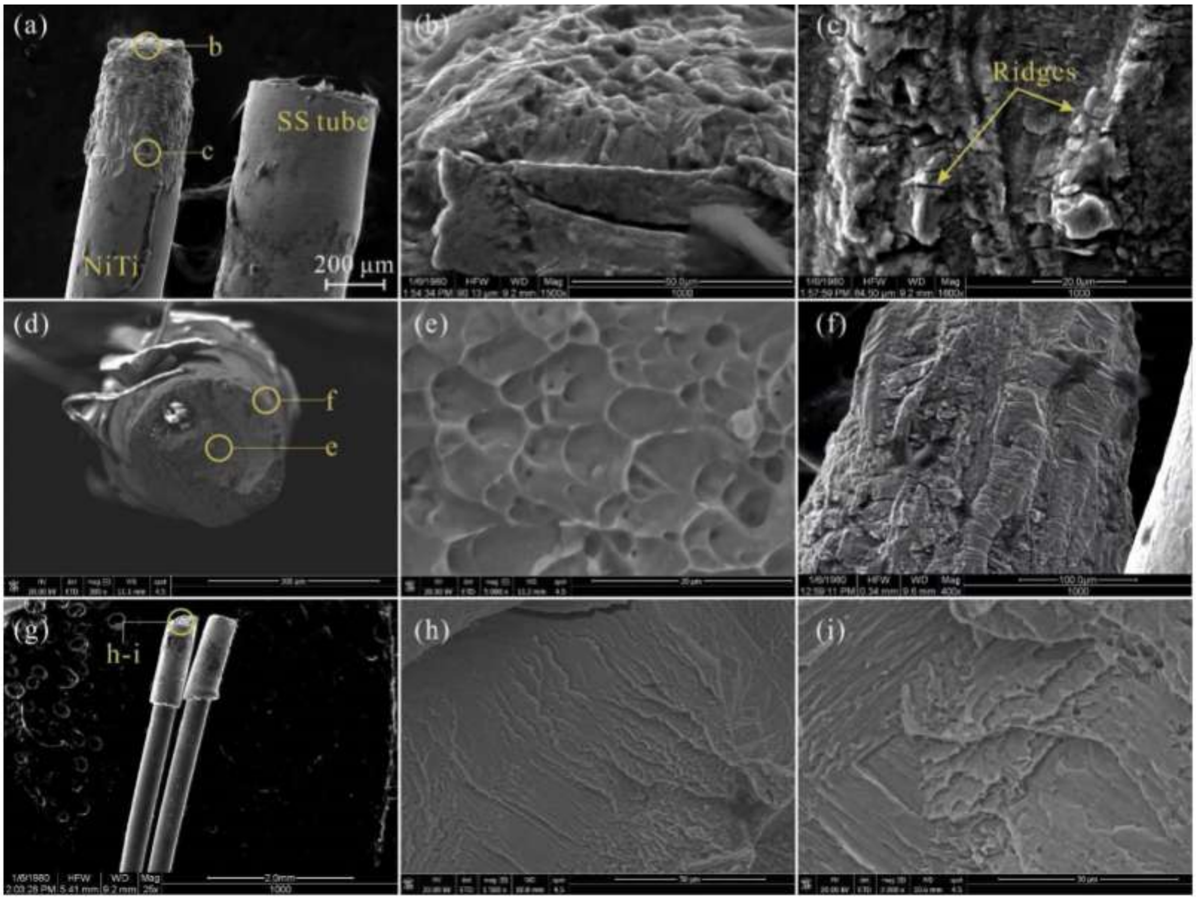

Figure 3: Fracture morphologies of NiTi/SS joints when welded

Mechanical Behavior

Mechanical Properties — Tensile Strength, Elongation, and Condition. The typical two conditions for Nitinol ultra-fine wire are cold worked and straight annealed. The preference depends on the processes of the medical device OEM, the specific requirements for the devices and their manufacturing operations being employed. Most of the wires will be heat set into a super-elastic condition in the next steps of the manufacturing process.

Controlling all these characteristics throughout the wire production and ensuring uniform dimensions is essential to producing good quality, reliable products and saving money in production. Precision equipment is required to retain these properties in the final product. Even minor inconsistencies can result in various issues, such as unusable parts or excess scrap, at the next manufacturing step in coil reinforcement.[16]

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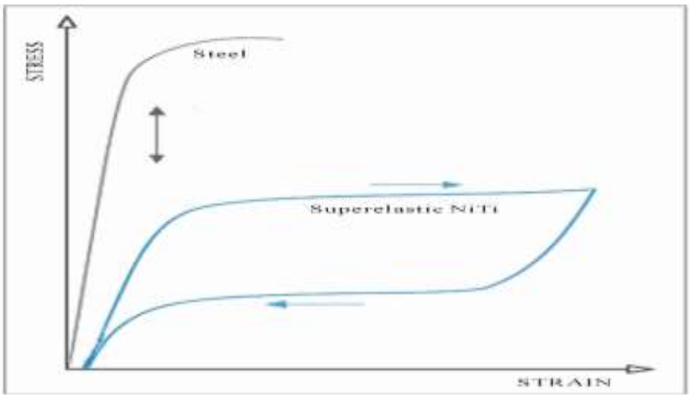
Stress-Strain Curve



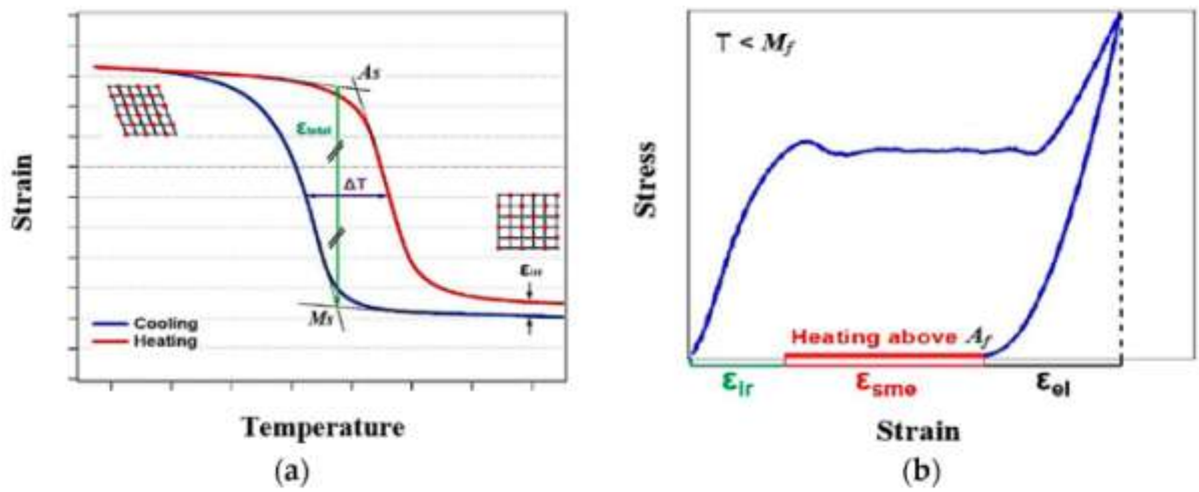


Figure 4

Failure Analysis

Nitinol, a near equiatomic intermetallic of nickel and titanium, is the most widely known and used shape memory alloy. Owing to its capacity to undergo a thermal or stress-induced martensitic phase transformation, Nitinol displays recoverable strains that are more than an order of magnitude greater than in traditional alloys, specifically as high as 10%. Since its discovery in the 1960s, Nitinol has been used for shape memory properties for couplings and actuators, although its contemporary use has been in medical devices. For these applications, stress -induced transformation ('super-elasticity') has been used extensively for self-expanding implantable devices such as endovascular stents and vena cava filters and tools such as endodontic files. Most of these applications involve varying biomechanical stresses or strains that drive the need to understand this alloy Nitinol's fatigue and fracture resistance fully. A near equiatomic intermetallic of nickel and titanium is the most widely known and used shape memory alloy. Owing to its capacity to undergo a thermal or stress-induced martensitic phase transformation, Nitinol displays recoverable strains that are more than an order of magnitude greater than in traditional alloys, specifically as high as 10%. Since its discovery in the 1960s, Nitinol has been used for shape memory properties for couplings and actuators, although its contemporary use has been in medical devices. For these applications, stress -induced transformation ('super-elasticity') has been used extensively for self-expanding implantable devices such as endovascular stents and vena cava filters and tools such as endodontic files. Most of these applications involve cyclically varying biomechanical stresses or strains that drive the need to fully understand this alloy Nitinol's fatigue and fracture resistance , a near equiatomic intermetallic of nickel and titanium, the most widely known and used shape memory alloy.

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As we know, Nitinol is an almost 1:1inter-metallic alloy metal of nickel and titanium; it is the most widely known and used shape memory alloy. Due to its ability to undergo a thermal or stress-induced martensitic phase transformation, Nitinol displays recoverable strains higher than almost all other traditional alloys, specifically with its capacity of recovery being as high as 10%. Nitinol has found various uses over time due to its shape memory properties for couplings and actuators in some of the earliest metal uses [6]. However, it has found its way into use for various medical devices[3]. For these applications, the stress-induced transformation, or the super-elasticity property of the metal, has been used extensively for self-expanding implantable devices such as endovascular stents and vena cava filters and tools such as endodontic files. Most of these applications involve cyclically varying biomechanical stresses or strains that drive the need to understand this alloy's fatigue and fracture resistance fully. [17]

Applications/Usage

Although it was initially developed, Nitinol was not considered a front-runner for the development of many new products and solutions in the bio-medical field. Mainly because since Nitinol is composed of almost 50% of nickel, a known allergen, nickel is also known to be a carcinogen. However, after several experiments were conducted to test for biocompatibility, it proved untrue in the case of Nitinol as the unique chemical bonding and the crystalline structure of the metal kept the nickel from forming any toxic effects was largely immune from the impact of corrosion as well. This allowed the medical industry to take advantage of its unique properties and develop new products that had not been possible before.

Another excellent application for Nitinol wire in the medical field is braided stents. Nitinol stents are becoming increasingly widely used in surgeries, especially to treat stenos is issues and issues below the knee. Nitinol stents would be used for the latter and is often a suitable choice for the treatment of peripheral vascular disease. This is due to the force Nitinol can provide when holding open vessels while still having enough elasticity to stretch and breathe. These two applications above are the primary use of Nitinol round-shaped wire, but some of the other applications also include:

- Heat engines
- Resilient glass frames
- Orthodontic arc wire
- Medical devices
- Actuators
- High-reliability couplings
- Temperature control system couplings

II. CONCLUSION

Over the last few decades, Nitinol has found itself at the center of numerous applications, especially in the medical field. It can adapt to extraordinary stresses and strains and is bio-compatible with the human body. What usually starts as ultra-fine wire on Nitinol is used for braid products such as stents, orthodontic wire, catheters, and other surgical implants and medical devices. With end uses like these, medical device manufacturers need to select an alloy and a supplier that provides materials with undeviating, consistent mechanical properties as well as strict quality-controlled methods of production in place, ensuring that those properties meet the requirements for processing medical wire while also remaining biocompatible over a long duration [3]. Nitinol has become an essential metal; many industries and many lifesaving and primary products are based on it. Both production methods and manufacturing sources are equally important in supply chains within the high-tech medical device industry. The future uses of Nitinol are many and can have a significant impact. Many potential benefits and applications of shape memory alloys ensure a bright future for these metals [6]. Research is currently being carried out on how to incorporate and use Nitinol in the field of robotics as well as newer engines and materials science fields. With the innovative ideas for applications of Shape Memory Alloys and the number of products on the market using Shape Memory Alloys continually growing, advances in the field of shape memory alloys for use in many different applications seem to have a very bright future indeed. Nitinol being such a versatile metal, its uses are constantly being explored. Over the next few years, its potential applications will only increase, thus making this unique metal a vital part of our lives. [6]

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