Design, Fabrication And Testing Of A Shape Memory Alloy Based Cryogenic Thermal Conduction Switch

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DESIGN, FABRICATION AND TESTING OF A SHAPE MEMORY ALLOY BASED CRYOGENIC THERMAL CONDUCTION SWITCH

by

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A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in the Department of Mechanical, Materials and Aerospace Engineering in the College of Engineering and Computer Science at the University of Central Florida Orlando, Florida

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2004

Advisor: Dr. Raj Vaidyanathan
ABSTRACT

Shape memory alloys (SMAs) can recover large strains (e.g., up to 8%) by undergoing a temperature-induced phase transformation. This strain recovery can occur against large forces, resulting in their use as actuators. The SMA elements in such actuators integrate both sensory and actuation functions. This is possible because SMAs can inherently sense a change in temperature and actuate by undergoing a shape change, associated with the temperature-induced phase transformation. The objective of this work is to develop an SMA based cryogenic thermal conduction switch for operation between dewars of liquid methane and liquid oxygen in a common bulk head arrangement for NASA. The design of the thermal conduction switch is based on a biased, two-way SMA actuator and utilizes a commercially available NiTi alloy as the SMA element to demonstrate the feasibility of this concept. This work describes the design from concept to implementation, addressing methodologies and issues encountered, including: a finite element based thermal analysis, various thermo-mechanical processes carried out on the NiTi SMA elements, and fabrication and testing of a prototype switch. Furthermore, recommendations for improvements and extension to NASA’s requirements are presented. Such a switch has potential application in variable thermal sinks to other cryogenic tanks for liquefaction, densification, and zero boil-off systems for advanced spaceport applications. The SMA thermal conduction switch offers the following advantages over the currently used gas gap and liquid gap thermal switches in the cryogenic range: (i) integrates both sensor and actuator elements thereby reducing the overall complexity, (ii) exhibits superior thermal isolation in the open state, and (iii) possesses high heat transfer ratios between the open and closed states. This work was supported
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Dedicated to My Parents
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LIST OF ABBREVIATIONS / NOMENCLATURE / ACRONYMS

$A_f$  
Austenite finish

$A_p$  
Austenite peak

$A_s$  
Austenite start

DFA  
Design For Assembly

EDM  
Electrical Discharge Machining

KSC  
Kennedy Space Center

$M_d$  
Martensite desist

MEMS  
Micro-Electro-Mechanical Systems

$M_f$  
Martensite finish

$M_p$  
Martensite peak

$M_s$  
Martensite start

NASA  
National Aeronautics and Space Administration

R-phase  
Rhombohedral-phase

SMA  
Shape Memory Alloy

Tank-M  
Experimental setup tank simulating methane dewar

Tank-O  
Experimental setup tank simulating oxygen dewar

TEM  
Transmission Electron Microscopy

TIG  
Tungsten Inert Gas
CHAPTER ONE: INTRODUCTION

1.1 Motivation

Shape memory alloy (SMA) actuators have remarkable potential for use in space applications due to the following advantages: (i) high power to weight and stroke length to weight ratios; (ii) combination of sensor and actuator elements in a single component; (iii) clean, debris-less, spark-free, silent operation and (iv) capability of operating in zero gravity environments with small, controlled accelerations.

The motivation for this project comes from NASA-KSC’s requirement for thermal management at cryogenic temperatures. The main objective of this project is to design, construct and test an SMA thermal conduction switch to facilitate thermal conduction of approximately 8 watts between two liquid reservoirs held at 118 K and 92 K. This switch would control the liquid methane temperature and pressure in a zero boil off system by allowing on-demand heat transfer between two reservoirs kept at separate temperatures, in an efficient and autonomous manner. Such a switch would support methane liquefaction for future Mars missions in addition to fulfilling immediate requirements at NASA-KSC.

The cryogenic range thermal switches that are currently used range from gas gap and liquid gap thermal switches that rely on convective heat transfer between two surfaces to externally mechanically actuated thermal switches. The sensors and active controls in such systems make them more complicated and expensive, yet less efficient than the proposed switch. Furthermore, the gas gap switches are restricted to long cycle times, tend to exhibit poor thermal
isolation in their open state, and have low heat transfer ratios between open and closed states. Other systems using conduction bands make use of mechanical means to generate sufficient thermal contact and may not be reliable. SMA thermal switches have the potential to limit these problems.

1.2 Shape Memory Alloys

“Shape memory alloys” (SMAs) or “memory metals” refer to a unique class of alloys that remember their original shape (or pre-deformation shape) when heated from some relatively low temperature. Heating results in a reversible phase transformation from a weaker to a stronger phase, followed by an associated recovery of all the accumulated strain. Generally, shape memory alloys can accommodate severe deformation (e.g., up to 8%) in their low-temperature, weaker phase. The strain recovery can take place against large forces, resulting in their application as actuators [Funakubo 1987, Schetky 1990, Uchino 1998, Vaidyanathan 2002]. Even though many alloys are found to exhibit the shape memory effect, only those alloys that recover considerable amount of strain (or exert significant force) are of prime importance. These include NiTi alloys and copper based alloys such as Cu-Al-Ni and Cu-Zn-Al.

In 1932 Arne Ölander, a Swedish physicist, first reported the thermoelastic transformation of martensite when working with an alloy of Au and Cd [Ölander, 1932]. Following his discovery, the shape memory phenomenon as a result of the thermoelastic behavior of the martensite phase was widely reported by Kurdjumov and Khandros [Kurdjumov & Khandros 1949] and Chang and Read [Chang & Read 1951]. In 1962, Buehler and his co-workers at the U.S. Naval Ordinance Laboratory discovered the shape memory effect in an
equiatomic alloy of Ni and Ti, which is considered a breakthrough in the field of SMAs [Buehler & Wang 1967]. This alloy was named Nitinol, acronym for “Nickel-Titanium Naval Ordinance Laboratory”. Since then, interest in the field of SMAs has increased immensely with significant advances in the understanding of SMA phenomena. More recently, progress has been made in magnetic SMAs. For example, O’Handley and co-workers showed in 1996 that a newly cut, polished and annealed single crystal of Ni-Mn-Ga showed a distinct kink when placed under a strong magnetic field [MPC Industry Collegium Report 1999]. The major properties of SMAs are discussed below.

1.2.1 Shape Memory Effect

The shape memory effect is the ability of an alloy to remember its pre-deformation shape when it is heated from some relatively low temperature. During this heating process, a reversible phase transformation takes place from the low-temperature, low-strength, martensitic phase to the high-temperature, high-strength, austenitic phase. All the accumulated strain is recovered during this phase transformation, and the recovery can take place against large forces [Funakubo 1987, Schetky 1990, Uchino 1998, Vaidyanathan 2002]. The shape memory effect can be of two types, namely, the one-way shape memory effect or the two-way shape memory effect. If the alloy remembers only its high-temperature shape, it is called the one-way shape memory effect. If in addition to the high-temperature shape the alloy remembers its low-temperature shape (deformed shape), it is called the two-way shape memory effect. This two-way shape memory effect is achieved by “biasing” or “training” the SMA specimen, where it is cycled several times between its low-temperature shape and high-temperature shape.
Figure 1.1: Transformation temperatures during shape memory effect

When an SMA in its low-temperature martensitic form is heated (Figure 1.1), the transformation from martensite to austenite begins at $A_s$ (austenitic start temperature). At $A_f$ (austenitic finish temperature), it completely transforms to austenite. Upon cooling (Figure 1.1), the reverse transformation back to martensite takes place at $M_s$ (martensitic start temperature) and at $M_f$ (martensitic finish temperature), all the austenite is completely transformed back to martensite [Buehler and Wang 1967]. Composition and thermo-mechanical treatments can significantly influence these transformation temperatures.
Figure 1.2: Lattice structure during shape memory effect

The mechanism behind the shape memory effect is deformation twinning in the martensitic phase followed by a thermally induced phase transformation to austenite (Figure 1.2). This thermally induced phase transformation is a first-order phase transformation that exists over a range of temperatures and the specific temperatures involved depend on the composition of the alloy being used, along with the history of cold working and metallurgical treatments performed. The specific changes that lead to the observed shape memory effect are due to a diffusionless transformation where the interatomic distances in the alloy are large compared to the displacements of the atoms.
1.2.2 Superelasticity

Another unique property of certain shape memory alloys is superelasticity or pseudoelasticity. Unlike the shape memory effect, superelastic behavior takes place without any temperature change, and this phenomenon is observed in a narrow range of temperatures just above the $A_f$ (austenitic finish temperature). Here large strains are possible during mechanical loading, due to the formation of stress-induced martensite (Figure 1.3). Unloading results in the conversion of unstable martensite back to austenite with an immediate recovery of all the accumulated macroscopic strain. At temperatures above $M_d$ (martensite desist), stress-induced martensite no longer forms (Figure 1.1).

1.2.3 Magnetic SMAs

Another class of SMAs is the magnetic SMAs, where the shape change is induced by a magnetic field. Freshly cut, polished and annealed single crystals of Ni-Mn-Ga show the appearance of twin boundaries (distinct kinks) when placed directly in a magnetic field. Here,
shear strains having a magnitude of 5% can be induced by a magnetic field of 4000 Oe at room temperature [MPC Industry Collegium Report 1999].

1.3 SMA Actuators

Figure 1.4: Types of SMA actuators (a) one-way actuator, (b) biased two-way actuator, and (c) differential two-way actuator

An SMA actuator is essentially a kind of thermal actuator that converts thermal energy into mechanical energy. It utilizes the shape memory effect to generate force and motion. SMA actuators act both as a temperature sensor and a work-generating element. When compared to electronically controlled thermal actuators comprising a temperature sensing device (e.g., thermistor), an electronic processor (e.g., IC circuit) and electric actuator (e.g., motor), SMA
actuators have fewer parts, higher reliability and lower fabrication costs. SMA actuators can be of one-way or two-way type (Figure 1.4).

One-way SMA actuators (Figure 1.4a) are used where one-time actuation is necessary such as in safety devices, couplings, etc. In a one-way actuator, the SMA element acts against some force when the temperature reaches a pre-set temperature. Two-way actuation can occur either in a biasing mode (Figure 1.4b) or a differential mode (Figure 1.4c). The biasing mode uses a bias spring opposing the SMA spring for actuation in either direction. When the temperature of the SMA element rises to a pre-set temperature, the SMA element being stronger as it undergoes a phase transformation, will act against the bias spring force actuating in one direction. As the temperature of the SMA element drops below a certain temperature, the bias spring force overcomes the SMA element force, thus acting in the opposite direction. Biasing mode operation is more often employed and offers enhanced flexibility in design. In the differential mode of operation, the bias spring is substituted by another SMA spring. Here actuation in either direction can be achieved by appropriately heating or cooling either of the two SMA elements. Applications requiring precise movement and high accuracy make use of the differential mode [Ohkata & Suzuki 1998].

NiTi is a favorite choice for designing actuators due to the following advantages: (i) able to accommodate large strains (up to 8%), (ii) high corrosion resistance [Buehler & Wang 1967, Funakubo 1987 and Duerig & Pelton 1994], (iii) capable of operating in a relatively wide range of temperatures by slightly varying the composition or thermo-mechanical treatments [Saburi 1988, Miyazaki et al.1986, Melton 1990, Mulder et al. 1993, Thoma et al. 1993], (iv) superior
1.4 SMA Actuators for Space Applications

Present-day space applications of SMA actuators can be found in deployable structures and light-weight, thermally controlled actuators in spacecrafts. The deployable structures are used to release solar cell panels, antennae, large frames etc. after a spacecraft launch. SMA-based deployable structures can be folded easily owing to their large martensitic deformability and perform the deployment scheme in a smooth movement when heated in a controlled manner and due to their inherent damping characteristics. Such SMA based deployable structures were used to deploy solar panels for NASA’s Hubble space telescope and to open the cover glass of a solar cell for NASA’s Mars Pathfinder Rover [AMT 2003, Landis & Jenkins 1997]. A thermally controlled SMA actuator is used in the gas analyzing instrument (Ptolemy) of the European Space Agency’s comet-chasing Rosetta spacecraft mission [ESA News 2004].

In general, SMA actuators are particularly advantageous for space applications in that [Krishnan et al. 2004]: (i) They combine sensory and actuation functions. The SMA element inherently senses a change in temperature and actuates by undergoing a shape change as a result of a phase transformation. Consequently, the need for external electronic sensors and control is eliminated. (ii) They function in a clean, debris-less, spark-free manner. The shape change that is responsible for the actuator displacement is again an inherent material property. It is not associated with moving parts that require lubrication or electrical signals with a potential to spark. (iii) They have high power/weight and stroke length/weight ratios. The operating range
includes strain and stress limits of 8% and 700 MPa, respectively, depending on the number of required cycles. (iv) They possess the ability to function in zero-gravity environments with small, controlled accelerations. The displacement strains are a result of a thermally induced phase transformation which can be controlled by the heat transfer rate. As a result, end-of-deployment shock loadings (associated with spring deployed structures) can be avoided in SMA-based deployable structures, thus eliminating the overall complexity by avoiding the use of dampers. (v) They are reliable and offer design flexibility. SMA actuators can function in a linear or rotary manner (or a combination of the two).

1.5 Organization

The work in this thesis is organized as follows: Chapter 2 explains the metallurgical aspects of NiTi SMAs relevant for actuator development and typical applications of NiTi SMAs. Chapter 3 discusses the design of the cryogenic thermal conduction switch from conceptual design to final design, addressing design issues, methodologies and intermediate designs. Chapter 4 discusses some fundamental aspects of cryogenic heat transfer and cryogenic heat transfer analyses performed on the SMA thermal conduction switch. Chapter 5 describes the thermo-mechanical processing and dilatometric measurements of SMA elements. Chapter 6 discusses the fabrication and testing procedure of prototypes. Chapter 7 presents the results, discussion and puts forth recommendations. Finally, Chapter 8 presents conclusions and lists potential applications of the thermal conduction switch. The design calculations, design drawings and actuation movie details are included in the Appendix.
CHAPTER TWO: LITERATURE REVIEW

Ever since the discovery of the shape memory effect in NiTi alloys, it has become the most extensively used SMA owing to the best combination of material properties they possess [Buehler & Wang 1967, Funakubo 1987 and Duerig & Pelton 1994]. This chapter is a review of metallurgical aspects of NiTi SMAs and their applications.

2.1 NiTi Shape Memory Alloys

Ordered intermetallic NiTi with near equiatomic composition forms the basis of all NiTi alloys. Close to room temperature, a near equiatomic NiTi undergoes a phase transformation between a monoclinic (B19'), martensite phase and a cubic (B2), parent austenite phase. Some NiTi alloy systems show a martensite-like transformation upon cooling, prior to the martensitic transformation, called an R-phase transformation. Here a transformation from the parent cubic phase (austenite) to a rhombohedral phase takes place.

NiTi alloys are thermally stable, wear resistant, corrosion resistant, and tolerate fairly large amounts of strain in the martensitic phase. The biocompatibility of NiTi alloys makes them a favorite for medical applications. When a NiTi element like a rod or spring in the austenitic phase is dropped on a hard surface, a sound wave propagates relatively unobstructed through the element, generating a “ringing sound”. In contrast, when an element in its martensitic phase is dropped, it generates a “thud sound”. This is because in martensitic NiTi the orientation of atoms acts as sound absorbing boundaries.
Depending on the composition (either deviating from equiatomic NiTi or substituting a small amount of Ni with another metal), history of cold working and history of heat treatments, the phase transformation in NiTi alloys exhibits complicated behavior. This may include one or more combinations of a shift in transformation temperatures, appearance of intermediate phases and changes in hysteresis. Consequently, the shape memory effect and the superelastic response in NiTi alloys are influenced.

2.1.1 Hysteresis in NiTi Alloys

The difference between transformation temperature ranges upon heating the martensite to the austenite (parent phase) and cooling the austenite back to the martensite is called the “transformation temperature hysteresis” (thermal hysteresis). Normally, hysteresis is defined as the difference between the temperatures at which the alloy is 50% transformed to austenite upon heating and 50% transformed to martensite upon cooling. Generally, thermal hysteresis ranges from 1.5-2 K for R-phase transformations, 10-15 K for certain Ni-Ti-Cu alloys, 20-60 K for binary NiTi alloys and around 100 K for Ni-Ti-Nb alloys [Suzuki & Horikawa 1991, Duerig & Pelton 1994].

The potential barrier against interface movement accounts for the origin of thermal hysteresis during martensitic transformations [Suzuki & Horikawa 1991]. In the usual polycrystalline form, the differences in crystallographic orientation among grains create different transformation conditions in each grain as opposed to a single crystal, single variant shape memory alloy that does not exhibit hysteresis between transformation temperatures [SmartLab
Aspects such as alloy composition, thermo-mechanical treatments, thermal cycling, pre-deformation and external load influence the transformation temperature hysteresis.

### 2.1.2 Phase Transformation Sequence in NiTi Alloys

The parent cubic (B2) phase may transform to any of three different crystal structures, i.e., the monoclinic (B19’) phase, the rhombohedral (R) phase or the orthorhombic (B19) phase, depending on various conditions. The phase transformation can follow different paths: $B2 \rightarrow B19'$, $B2 \rightarrow R \rightarrow B19'$, $B2 \rightarrow B19 \rightarrow B19'$ and $B2 \rightarrow R \rightarrow B19 \rightarrow B19'$ [Beyer, 1995]. The conditions determining the above paths are [Miyazaki & Otsuka 1989]: (i) variation of Ni content [Melton 1990], (ii) addition of alloy elements [Khachin 1989, Nam et al. 1990, Saburi 1993], (iii) ageing after solution treatment [Nishida & Honma 1984, Honma 1987, Saburi 1988], (iv) thermo-mechanical treatments [Mulder et al. 1993, Thoma et al. 1993], and (v) thermal cycling [Miyazaki et al. 1986, Mulder et al. 1994]. Also, it has been mentioned that under certain conditions the B2 phase changes to an incommensurate phase and subsequently transforms to the R phase [Folkins & Walker 1989, Beyer 1995]. The so-called transformation to the incommensurate phase is not really a phase change, but shows satellite deviations (in the electron diffraction pattern) without altering the lattice structure. The model suggested by Folkins and Walker assumes the satellites correspond to sinusoidal modulations of the parent cubic (B2) phase.
2.1.2.1 **R-phase in NiTi Alloys**

The R-phase transformation is coupled with a temperature hysteresis as low as 1.5 K (compared to a hysteresis in excess of 10 K for a martensitic transformation). Additionally, the R-phase has a significant contribution to the two-way shape memory effect [Mo *et al.* 1986, Zuyao *et al.* 1988]. Due to very low transformation hysteresis, they are useful in designing certain actuators. By restricting the temperatures around the region of transformation between the parent cubic phase and the R-phase, one can observe both the shape memory effect and superelasticity. The lattice distortion (cubic to rhombohedral) and consequently the recoverable strain increases with decrease in temperature. However, the maximum recoverable strain (~1%) is fairly low compared to the martensitic shape memory effect [Otsuka, 1990]. Furthermore, the fatigue life for the R-phase is very good compared to the martensite phase [Tamura *et al.* 1986].

The formation of the R-phase is attributed to the presence of coherent particles in the alloy matrix [Ren *et al.* 1999, Khalil Allafi *et al.* 2002]. The presence of particles produces a strong resistance to large lattice variant transformation associated with the formation of B19’. A transformation through the R-phase produces a significantly small lattice variant transformation and is much less affected by particles [Ren *et al.* 1999]. The R-phase can be achieved by suppressing the martensitic transformation, i.e, by: (i) increasing the Ni content, (ii) ageing at lower temperatures (573-773 K) after solution treatment, (iii) annealing below the recrystallization temperature immediately after cold-working, and (iv) adding a third element such as Fe or Co [Saburi 1988, Miyazaki *et al.* 1986].
2.1.3 Mechanical Properties of NiTi Alloys

Table 2.1: Mechanical properties of NiTi [Hodgson, 1988]

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<thead>
<tr>
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<th>Young’s Modulus</th>
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<td></td>
<td>Austenitic</td>
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<td></td>
<td>Martensitic</td>
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<td>Yield Strength</td>
<td>Austenitic</td>
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<td>Martensitic</td>
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<tr>
<td>Ultimate Tensile Strength</td>
<td>Fully Annealed</td>
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<td></td>
<td>Work Hardened</td>
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<tr>
<td>Poisson’s Ratio</td>
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</tbody>
</table>

NiTi based alloys have superior mechanical properties compared to other SMA alloys. Their tensile strength is as high as 895 MPa, exhibit up to 50 - 60% elongation at failure and can withstand 8% strain in the martensitic form (that can be fully recovered on heating). They have outstanding mechanical properties among intermetallics and can be used as a structural material. They are exceptionally resistant to abrasion and corrosion. Similar to most other SMAs, NiTi alloys exhibit marked differences in their mechanical behavior depending on their austenitic or martensitic phase. Table 2.1 shows the mechanical properties of austenitic and martensitic NiTi.
Figure 2.1: Stress-strain curves of NiTi in austenitic and martensitic states.

Figure 2.1 shows the general stress-strain characteristics of NiTi in the austenitic and martensitic states. When stress is applied to NiTi in the martensitic state, initially under low stresses the twinned lattice will deform elastically up to the martensitic yield point. As the load increases, martensitic deformation takes place and the rest of the martensitic stress-strain curve can be divided into three distinct regions [Perkins 1974, Mohamed & Washburn 1977, Melton & Mercier 1978]. Deformation twinning results in the formation of an initial low plateau region in the stress-strain curve. A second region that is usually linear follows this (not purely elastic). This second region is assumed [Melton & Mercier 1978] to be a mixture of elastic deformation of the detwinned martensite, along with the formation of new orientations of martensite that intersect those already present. The first and second regions constitute heat recoverable strain.
(represented by point A in Figure 2.1). The third region is similar to yielding in regular metals, as a result of the inception of irreversible plastic deformation.

Table 2.2: Fatigue properties of NiTi [Stöckel, 1992]

<table>
<thead>
<tr>
<th>Cycles</th>
<th>Max. Strain</th>
<th>Max. Stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8%</td>
<td>500 MPa</td>
</tr>
<tr>
<td>100</td>
<td>4 %</td>
<td>275 MPa</td>
</tr>
<tr>
<td>10000</td>
<td>2 %</td>
<td>140 MPa</td>
</tr>
<tr>
<td>100000+</td>
<td>1 %</td>
<td>70 MPa</td>
</tr>
</tbody>
</table>

Table 2.2 by D. Stöckel in 1992 shows the fatigue properties of typical binary NiTi alloys. Generally, NiTi alloys do extremely well in low-cycle, strain controlled environments, and do relatively below par in high-cycle, stress-controlled environments [Duerig & Pelton 1994]. However, it should be realized that ternary alloys such as Ni-Ti-Cu or special treatments could yield much higher values of tolerable maximum strains and stresses.

### 2.1.4 Physical Properties of NiTi Alloys

Table 2.3 shows the physical properties of austenitic and martensitic NiTi such as the melting point, density, thermal conductivity, coefficient of thermal expansion and specific heat.
Table 2.3: Physical properties of NiTi [Hodgson, 1988]

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Point</td>
<td>1573K</td>
</tr>
<tr>
<td>Density</td>
<td>6.45 g/cm³</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td></td>
</tr>
<tr>
<td>Austenitic</td>
<td>0.18 W/cm-K</td>
</tr>
<tr>
<td>Martensitic</td>
<td>0.086 W/cm-K</td>
</tr>
<tr>
<td>Coefficient of thermal expansion</td>
<td></td>
</tr>
<tr>
<td>Austenitic</td>
<td>11.0 x 10^-6 / K</td>
</tr>
<tr>
<td>Martensitic</td>
<td>7.0 x 10^-6 / K</td>
</tr>
<tr>
<td>Specific Heat</td>
<td>0.20 cal/g-K</td>
</tr>
</tbody>
</table>

2.1.5 Effect of Grain Size in NiTi Alloys

Reduction in grain size appears to improve superelasticity and other mechanical properties [Beyer 1995].

2.1.6 Effect of Thermo-Mechanical Treatments in NiTi Alloys

Moderate cold working, followed by annealing below recrystallization temperatures, of NiTi alloys leads to a reduction in transformation hysteresis. NiTi when heavily cold-worked eliminates the martensitic plateau on the stress-stain curve and exhibits poor shape memory. This is because high densities of random dislocations are introduced, that obstruct the mobility of twin boundaries [Melton, 1990]. However, the yield strength is considerably improved during cold-working. Annealing restores shape memory, but decreases yield strength.

NiTi alloys are sensitive to temperature and time in a heat treatment process, owing to compositional changes that takes place in them. The composition can vary between TiNi, Ti₁₁Ni₁₄, Ti₂Ni₃ and TiNi₃, by varying either temperature or time [Beyer, 1995]. Additionally,
grain size and defect structures change with the type of heat treatment. For example, increasing heat treatment time can decrease grain size and defects.

When Ni-rich alloys are aged at lower temperatures (less than 773 K) or slowly cooled after a heat treatment, the alloy decomposes to a more Ti-rich matrix with a Ni-rich phase finely dispersed in it [Melton, 1990]. This causes an elevation in transformation temperatures. Hence, aging of Ni-rich alloys are done at 773-973 K to preserve the lower transformation temperatures. Additionally, ageing at higher temperatures suppresses the R-phase transformation and superelasticity in Ni-rich binary alloys. The instability occurs on the Ni-rich side because of the solubility range of NiTi extending to higher Ni contents at temperatures above around 773 K [Wasilewski, 1974]. However, for Ti-rich compositions there is very little variation involving heat treatment conditions [Melton, 1990].

2.1.7 Effect of Thermal Cycling in NiTi Alloys

Thermal cycling of solution treated NiTi alloys (without ageing) results in a gradual shift of transformation temperatures after each cycle. However, the transformation temperatures remain constant for aged Ni-rich alloys and those annealed at temperatures lower than the recrystallization temperature after cold-working [Miyazaki et al. 1986].

2.1.8 Effect of Deviation from Equiatomic Stoichiometry in Binary NiTi Alloys

The stoichiometric range of NiTi is very narrow at low temperatures. Hence, at low temperatures the alloy tends to form precipitates of a secondary intermetallic phase when there is
a stoichiometric deviation from equiatomic NiTi. The microstructure will thus contain small amounts of secondary phases distributed in the matrix.

\( M_s \) depends fairly strongly on the composition, especially for Ni rich alloys. On the other hand, Ti rich alloys exhibit less sensitivity, mainly because of the formation of Ti rich precipitates, leaving the matrix composition essentially the same [Melton, 1990].

Increasing Ni content strongly depresses the transformation temperature. NiTi alloys exhibit superelasticity or pseudo-elasticity when the Ni content is above 50.6 at%. The effects of thermal cycling and annealing are substantial when the Ni content is high [Honma 1987]. Furthermore, excess Ni increases the yield strength of the austenite.

### 2.1.9 Effect of Alloy Element Additives in NiTi Alloys

NiTi is fairly sensitive to alloy element additives. Addition of Fe, Al, Cr, Co and V tend to substitute Ni and depresses the \( M_s \) [Goldstein et al. 1964, Chernov et al. 1979]. The depression of \( M_s \) is strongest with the addition of Cr and weakest with V and Co. Their additions have practical importance in creating cryogenic SMAs, stiffening a superelastic alloy, or increasing the separation of the R-phase from the martensite phase [Duerig & Pelton 1994].

Addition of Nb and Cu alters the hysteresis and martensitic strength. Hysteresis is increased with the addition of Nb, while with Cu [Melton & Mercier 1978] it is reduced. Cu can substitute Ni for up to 30 atomic % without any changes in the shape memory effect. Additionally, addition of Cu causes two step transformations, cubic(B2) \( \rightarrow \) orthorhombic(B19) \( \rightarrow \) monoclinic(B19’) [Moberly & Melton 1990, Saburi 1993, Nam et al. 1990]. The effect of substitution of Cu makes the transformation temperature less sensitive to the Ni content, in
addition to reducing the hysteresis to about 15°C. Most benefits of adding Cu are shown only up to 10% and extra additions show only marginal improvements.

### 2.1.10 Fabrication Techniques for NiTi Alloys

Due to the reactivity of Ti and its tendency to oxidize, NiTi alloys are made from their constituents by melting in vacuum or inert gas. Vacuum induction melting and vacuum arc melting are the most common methods employed. For certain commercial applications they are made by double vacuum melting, first by vacuum induction melting and then by vacuum arc melting to enhance homogeneity and structure of the alloy [Schetky & Wu 2003]. Sintering by Hot Isostatic Pressing (HIP) can make relatively dense NiTi alloys.

The hot workability of NiTi is moderately good, while cold workability is difficult due to rapid work hardening. Extrusion, rolling, forging and various forming operations of NiTi alloys are successfully employed commercially. However, their parameters remain proprietary. Machinability of NiTi is difficult; a very low cutting speed and lots of coolant is required for machining. In certain cases unconventional techniques such as abrasive machining or Electrical Discharge Machining (EDM) are more preferable.

Tungsten Inert Gas (TIG) or resistance welding methods can be used with relative ease to join NiTi to itself. Joining NiTi with other metals or alloys is difficult due to the fact that the mating metals or alloys cannot withstand large strains produced by SMAs [Duerig & Pelton 1994]. However, various other proprietary methods are employed to join in large-scale production.
2.2 **Applications of NiTi Shape Memory Alloys**

The history of application of NiTi SMAs can be traced back to the late 1960’s when SMA couplings ("Cryofit® couplings") were successfully used to join the high-pressure hydraulic system piping on Grumman F-14 fighter airplanes. The application of Nitinol in the medical field was first reported in the early 1970s [Cutright et al. 1973]. However, other large-scale applications were not followed until the introduction of SMA based mobile telephone antennae, eyeglass frames and brassiere underwires. Most current applications of SMAs can be categorized into three groups, based on their unique properties namely: (i) shape memory effect, (ii) superelasticity and (iii) martensitic deformability.

2.2.1 **Applications based on the Shape Memory Effect**

**Pipe Couplings:** NiTi alloy couplings are used to join hydraulic tubing in certain jet aircraft. The couplings, having sealing bands on the inside surface are machined in the austenitic condition with the inside diameter slightly less than the outside diameter of the pipes to be joined. They are then cooled to the martensitic condition using liquid nitrogen and the inside diameter is mechanically expanded slightly larger than the outside diameter of the pipes to be joined. This facilitates easy mounting to join the pipes or tubes. They are stored in liquid nitrogen until ready to install. Once installed, they shrink to fit tightly during warming back to ambient temperature. More recently, Ni-Ti-Nb alloys with wide thermal hysteresis eliminate the need for storage in liquid nitrogen.
**Muscle Wires:** SMA wires replicate biological muscle fibers. The actuation of these muscle wires can be made either by heating them externally or by heating them internally by passing electric current. This makes them suitable for use in robots and toys.

**Electrical Connectors:** Electrical connectors made of NiTi alloys provide a secure connection at ambient temperatures and can be easily released by cooling.

**Actuators:** SMA actuators integrate both sensory and actuation functions. They are commonly used as flap control for controlling airflow in air-conditioners, as a thermal switch for controlling coolant in automobiles, as an air damper for multi-function electric ovens, and as a thermal mixing valve in faucets. They are also used in various aerospace related applications such as deploying solar panels in the Hubble space telescope.

**Adaptive Wings:** Shape memory elements can contract when they are converted to the austenitic phase upon heating by an electric current and can expand when they become martensitic upon cooling by the surrounding air (when the current is switched off). This principle is used in adaptive aerofoils that change shape to minimize the drag of the aerofoil under the constraint of a constant lifting force.
2.2.2 Applications based on Superelasticity

**Eyeglass Frames**: Eyeglass frames made of superelastic NiTi can withstand severe deformation and spring back completely. The corrosion resistance and endurance of NiTi make it suitable for use under chemical and corrosive environments.

**Clothing**: Superelastic NiTi is being used as brassiere underwire for improving the body contour, as the core wire of petticoats that enhance the wedding gown’s appearance, in the rim of hats to retain their shape and in the counter (top of the heel) of shoes to protect them from wearing out [Furukawa 2004].

**Mobile Telephone Antennae**: The spring back capability of superelastic SMAs is desirable for withstanding possible deformation in mobile telephone antennae.

**Medical Guide Wires**: The biocompatibility of NiTi alloys along with spring back capability makes them unique for use as guide-wires and probes during surgery.
**Vascular Stents:** Vascular stents (Figure 2.2) can be used to hold human arteries open. Cylindrical vascular stents made of superelastic NiTi wires are compressed and inserted into the proper location in the artery. Upon releasing, they return to the original shape, reinforcing the walls of the artery.

**Orthodontic Wire:** Orthodontic wires are used to correct irregular teeth lines. NiTi alloy wires are superior to conventional stainless steel wire because of their constant correcting force and the large amount of correction they deliver.

### 2.2.3 Applications based on Martensitic Deformability

**Vibration Dampers:** Vibration dampers are used in the form of engine mounts and actuators for buildings.

**Structural Dampers:** Structural dampers are used to lessen the damage caused by an earthquake.

**Bendable Surgical Tools:** The handles of open-heart surgery tools made of martensitic NiTi allow them to be bent precisely to a suitable shape required by the surgeon during a surgery. They regain their original shape while heating for sterilization.
CHAPTER THREE: DESIGN OF THE THERMAL CONDUCTION SWITCH

3.1 Concept

![Figure 3.1: A schematic illustrating the concept for a shape memory alloy based cryogenic thermal switch; left – open position and right – closed position](image)

The initial design concept of the shape memory alloy based cryogenic thermal conduction switch for a NASA requirement of 8 watts thermal conduction between liquid methane reservoir (high-temperature reservoir) kept at 118 K and liquid oxygen reservoir (low-temperature reservoir) kept at 92 K is shown in Figure 3.1. The design uses a bias spring in addition to an SMA spring for two-way actuation. The SMA spring (liquid methane side) and bias spring are attached on either side of a moving copper contact. A flexible copper strap that is thermally connected to the moving copper contact is expected to provide a path for heat conduction when the switch is in the closed position (when the moving copper contact comes in thermal contact)
with the stationary copper contact on the liquid methane side). The SMA spring, sensing the temperature in the liquid methane tank, will undergo a phase transformation (from martensite to austenite) when the temperature in the liquid methane tank increases above a certain temperature. This phase transformation will be coupled with an increase in its strength that provides a thermal contact between the two copper contacts (moving copper contact and stationary copper contact on the liquid methane side) by working against the bias spring. Indium foils are used between the copper contacts to limit the variation in thermal conductivity with contact force. The conductive heat path thus established through the flexible copper strap results in liquid oxygen cooling liquid methane. When the temperature of the liquid methane tank drops to the specified temperature, the SMA spring will undergo a reverse transformation. This resulting weaker phase (martensite) will not be able to hold the bias spring, thus creating an open circuit for heat conduction.

3.2 Design Challenges and Issues

Droney et al. in 2003, developed a prototype during their senior design project that successfully demonstrated the feasibility of the concept in a switch that operated between ice water (273 K) and hot water (338 K) using commercially available NiTi. However, the implementation of the concept in a working prototype at cryogenic temperatures offers the following challenges:

1) No information is available for designing a biased two-way SMA actuator working in the cryogenic range.
2) Given that the application specific cryogenic NiTi alloys are not commercially available, the SMA elements should be chosen from a commercially available material, which has the lowest possible transformation temperatures. This offers fewer choices in design.

3) To match NASA’s requirements considering cryogenic and vacuum conditions. That is, to develop a prototype that would simulate heat transfer requirements between liquid methane dewar kept at 118 K and liquid oxygen dewar kept at 92 K.

The various issues that should be addressed in the design of the thermal conduction switch are:

1) The switch is expected to work in vacuum conditions, and quantifying the associated contact thermal resistance is not trivial.

2) Hysteresis in the commercial SMA element can result in a lag between switching temperatures.

3) A temperature gradient can develop over the length of the SMA element when the switch is in the closed condition, resulting in a non-uniform phase transformation over the length of the SMA element.

4) Adequate contact force has to be generated by the SMA element for effective conductive heat transfer.

3.3 Evolution from Initial Concept - Intermediate Designs

In the initial concept, the SMA and the bias spring are on either side of the moving copper contact that is attached to a flexible copper strap. When the switch is in the closed
position, a temperature gradient of 28 K (120 K – 92 K) would be distributed along the heat conduction length. This implies that the design offers only around 14 K between transformation temperatures for shape memory actuation. Taking into consideration the typically large transformation hysteresis in commercially available SMAs, a better option would be to position both the SMA element and the bias spring on the same side (methane side) of the moving copper contact. However, for the symmetry of actuation, the bias spring should be concentric to the SMA spring (unfeasible when accounting for “design for assembly” considerations as described in section 3.4) or there should be two (or more) SMA springs acting against a single bias spring as shown in Figure 3.2. The SMA springs are placed concentric to the support rods that support the moving copper plate (contact) and keep it in proper alignment.

Another concern is with the copper strap that provides the heat conduction path in the original concept. Calculations (see Appendix A) show that the heat transfer area required for a conduction path of 50 mm at cryogenic conditions is around 230 mm². This means that a total of 9 copper straps that are reasonably flexible (0.508 mm thick x 50.8 mm wide) would be required. A solution would be to provide an L-shaped copper bar as shown in Figure 3.2. The perpendicular section of the L-bar helps in closing the heat conduction circuit when the SMA spring (austenitic condition) pushes the moving copper plate towards the straight copper bar at the liquid oxygen end.
However there is a problem with the intermediate design I. When the SMA spring in the austenitic state closes up the gap between the copper contacts, thereby providing a heat conduction path, a temperature gradient sets up within the SMA spring. This temperature gradient is due to the fact that one end of the SMA spring is attached to the copper plate on the methane tank side and the other end is close to the oxygen tank temperature. Consequently, the
phase change will not be uniform within the SMA spring during heat transfer and also when the temperature of the liquid methane tank drops to the required specific temperature.

The intermediate design II (Figure 3.3) provides a solution to the problem of temperature gradient within the SMA spring. Instead of an L-shaped copper bar (conduction path) in the intermediate design I, an F-shaped copper bar is used. Additionally, the stationary copper plate
joined to the methane tank is insulated so that the heat transfer to the SMA element takes place only through the moving copper contacts.

3.4 **Design for Assembly Considerations**

Design for Assembly (DFA) is a design philosophy to lower total product cost by investigating the assembly process, assembly time and part cost of the product design in the early stages of development. Further DFA analysis will help to improve the reliability, serviceability and quality of the end product. One of the significant outcomes of DFA is reduction in the number of parts required for assembly. Some major DFA principles are: (i) minimize part count, (ii) encourage modular assembly, (iii) design parts with self-fastening/self-locating features, (iv) use of standard parts, (v) stack assemblies, (vi) eliminate reorientation, (vii) facilitate parts handling and (viii) minimize levels of assembly [Connelly et al. 1998].

Investigation of intermediate design II using DFA shows that the F-shaped copper conduction path is difficult to assemble/weld to the methane dewar, ensuring proper alignment and parallelism with the moving copper contact. Exploring alternatives that would solve the above mentioned problem and also reduce the number of parts, revealed the possibility of support rods carrying the functions of both heat transfer and alignment.
3.5 Final Design Model

Figure 3.4: Final design (model I)

Figure 3.5: Bias spring

Figure 3.4 shows the final design (model I) that was arrived by using helical springs as SMA elements. The design consists of a stationary plate (fixed copper contact) on the methane side, three support rods for support and heat transfer, three SMA helical compression springs concentric to the support rods, three spring seats that keep the SMA springs in position and also
insulate them from the stationary plate, a bias extension spring placed in between the SMA springs with bias spring holders on both ends, a moving plate (moving copper contact) for actuation, and three bushings fixed to the moving plate for easy sliding on support rods.

The two ends of the bias spring (Figure 3.5) were intentionally made straight due to dimensional constraints and for ease of adjustment of the distance between the two plates (stationary and moving plates). The bias spring holders were devised to adjust the length of the bias spring, with two set screws securing the ends of the bias spring.

### 3.5.1 Materials Selection

All the materials for the final design were selected by keeping in mind issues of low-temperature embrittlement and thermal conductivity. These materials included oxygen free pure Cu for the stationary and moving plates, Be-Cu alloys for support rods and bushings, austenitic stainless steels containing more than 7% Ni for bias spring, brass for bias spring holders and polytetrafluoroethylene (Teflon) for spring seats. Indium foil and Apiezon® N grease were used to enhance the thermal conductivity between mating parts.

### 3.5.2 SMA Spring Design

The SMA spring was designed using the following formulae [Waram 1990, Ohkata & Suzuki 1998]:

Stress concentration factor is given by:

\[
K = [(4C-1) / (4C-4)] + (0.615 / C)
\]
Shear stress is given by:
\[
\tau = \frac{(8 \ P \ D \ K)}{(\pi \ d^3)}
\]
\[
= \frac{(8 \ P \ C \ K)}{(\pi \ d^2)}
\]

Allowable shear strain is given by:
\[
\gamma_S = \gamma_{\text{max}} - \gamma_H ; (\gamma_{\text{max}} = 1\%)
\]

Number of turns is calculated by:
\[
n = \frac{\delta \ d}{(\pi \ \gamma_S \ D^2)}
\]

where, \( C \) = spring index, \( P \) = load on spring, \( \gamma_H \) = austenitic shear strain, \( D \) = mean diameter of coil, \( d \) = diameter of wire, and \( \delta \) = maximum deflection.

Using the above formulae, Table 3.1 shows the parameters derived for the SMA helical springs (see Appendix A for calculations).

<table>
<thead>
<tr>
<th>Load on each spring</th>
<th>16 N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deflection</td>
<td>10 mm</td>
</tr>
<tr>
<td>Spring index</td>
<td>12</td>
</tr>
<tr>
<td>Wire diameter</td>
<td>2.159 mm</td>
</tr>
<tr>
<td>Mean coil diameter</td>
<td>25.908 mm</td>
</tr>
<tr>
<td>Number of turns</td>
<td>4 (with ground ends)</td>
</tr>
<tr>
<td>Free length of the spring</td>
<td>23.636 mm</td>
</tr>
</tbody>
</table>
3.5.3 Bias Spring Design

Using helical spring theory, Table 3.2 shows the parameters derived for the bias spring (see Appendix A for calculations).

Table 3.2: Bias spring design parameters

<table>
<thead>
<tr>
<th>Material for bias spring</th>
<th>Stainless steel AISI 302</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load on the spring</td>
<td>20 N</td>
</tr>
<tr>
<td>Deflection</td>
<td>8 mm</td>
</tr>
<tr>
<td>Spring index</td>
<td>10</td>
</tr>
<tr>
<td>Wire diameter</td>
<td>1.628 mm (AWG 14)</td>
</tr>
<tr>
<td>Mean coil diameter</td>
<td>16.28 mm</td>
</tr>
<tr>
<td>Number of turns</td>
<td>6 (with straight ends aligned on a single axis)</td>
</tr>
<tr>
<td>Free length of the spring</td>
<td>9.768 mm</td>
</tr>
</tbody>
</table>

3.5.4 Force vs. Deflection of Bias and SMA Springs

The SMA spring was tested for stiffness after heat treatments (discussed in Chapter 5) under austenitic (room temperature) and martensitic (liquid nitrogen temperatures) conditions. The following spring rates were derived after testing:

High-temperature (austenitic) spring rate, $K_H = 1.7180 \text{ N/mm}$

Low-temperature (martensitic) spring rate, $K_L = 0.2108 \text{ N/mm}$
Figure 3.6 shows the force versus deflection of the bias spring (1/3\textsuperscript{rd} actual stiffness) and the SMA spring under austenitic and martensitic conditions (see Appendix A for calculations). The bias force is one-third that of the original stiffness of the bias spring because a single bias spring opposes three SMA springs in the final design. Since the bias spring opposes the SMA springs; hence their slopes have opposite signs. It is projected from the graph that the bias spring would be extended to around 10 mm by the SMA spring in the austenitic condition than in the martensitic condition. Thus, if the switch is compressed during assembly by around 5 mm in hot conditions, a contact force of about 36 N can be generated.
CHAPTER FOUR: CRYOGENIC HEAT TRANSFER ANALYSIS

4.1 Cryogenic Heat Transfer

Heat transfer at cryogenic conditions poses problems and limitations that are rarely associated with heat transfer at relatively higher temperatures. For example, most material constants that are applied to relatively short ranges of temperatures in standard heat transfer problems become variables at cryogenic temperatures. For example, a piece of aluminum when cooled from 20 K to 18 K (10% decrease) would experience a 37 % decrease in its specific heat [Barron, 1999].

4.2 Issues in Cryogenic Heat Transfer

Some of the major issues encountered in cryogenic systems are contact resistance, heat transfer through extended surfaces or fins and frost and, cool down with cryogenic fluid transfer systems and storage vessels [Barron, 1999]. However, since the proposed thermal switch is expected to work under vacuum conditions, only issues regarding contact resistance directly affect the design.

4.2.1 Contact Resistance

Thermal contact resistance is one of the major problems encountered in cryogenic systems. When heat is transferred between two solid surfaces that are brought in contact with
each other, a thermal resistance is developed at the interface and causes a temperature difference across the interface. In cases where a high heat flux is required, this temperature difference developed across the interface is detrimental. For cryogenic conduction under vacuum conditions, predicting the contact resistance is rather complicated.

Roughness and flatness deviations of the two surfaces in contact are the primary reasons for contact resistance. The heat transfer across the interface can be improved by either placing a soft metal foil like indium foil in between the contact surfaces or by applying thermal greases such as Apiezon® to the surfaces. However, when the contact surfaces are of moving or make and break types, application of most thermal greases are not recommended due to the fact that they solidify at cryogenic temperatures.

4.3 **Heat Transfer Requirements for Methane and Oxygen Dewars**

The proposed thermal switch is to work between methane and oxygen dewars, each with a capacity of 25 L. Considering the density and specific heat of methane to be 424 kg·m⁻³ and 3530 J·kg⁻¹·K⁻¹ at 118 K respectively, a 2 K rise in temperature will require 74.836 kJ of heat. Considering a heat leakage of 2W, this would take 10.394 hrs (see Appendix A for calculations).

With only a single switch of 8 kW capacity working between the dewars, it would take 3.465 hrs (6 kW effective cooling capacity) to cool down the methane dewar from 120 K to 118 K (see Appendix A for calculations).
4.4 Thermal Analysis using I-DEAS

The heat transfer analysis was carried out using TMG simulations of I-DEAS Master Series version 9 (Structural Dynamics Research Corporation). Both steady state and transient analyses were carried out. The objectives of the steady state analysis were to determine the temperature profile of the SMA element and the moving thermal contact, when the switch is in the closed position and the temperature of the methane tank is at 120 K. A transient analysis was carried out to study the response time.
An assembly model (Figure 4.1) was constructed to represent the actual geometry of the prototype. Finite element meshing was applied to the relevant contact surfaces and on the volume that represents geometrical symmetry of the model. Following are the assumptions made: (i) The initial temperature of the assembly is between 119 K and 120 K, (ii) the stationary copper contact is in equilibrium with the methane tank, and (iii) effects of convection and radiation are negligible. A set of boundary conditions (methane tank at 120 K and oxygen tank at 92 K) and thermal properties of the materials at 100 K (for those materials where data was available) were applied to the model.

### 4.4.1 Parameters

#### Table 4.1: Thermal properties of materials used in the model

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal Conductivity (W/m·K)</th>
<th>Specific Heat (J/kg·K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen Free Copper (100 K)</td>
<td>401</td>
<td>255.27</td>
</tr>
<tr>
<td>Beryllium Copper (100 K)</td>
<td>41.409</td>
<td>255.27</td>
</tr>
<tr>
<td>NiTi</td>
<td>18</td>
<td>320</td>
</tr>
<tr>
<td>Teflon</td>
<td>0.195</td>
<td>233.88</td>
</tr>
</tbody>
</table>

#### Table 4.2: Contact resistance of mating surfaces in the model

<table>
<thead>
<tr>
<th>Mating Surfaces</th>
<th>Contact Resistance (K/W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Support rods with stationary plate</td>
<td>0.0892</td>
</tr>
<tr>
<td>Bushings with moving plate</td>
<td>0.0509</td>
</tr>
<tr>
<td>Bushing with support rod</td>
<td>0.134</td>
</tr>
<tr>
<td>Spring seat with stationary plate</td>
<td>0.0444</td>
</tr>
<tr>
<td>NiTi spring with bushing</td>
<td>0.005540</td>
</tr>
<tr>
<td>Spring seat with NiTi spring</td>
<td>3</td>
</tr>
</tbody>
</table>
Due to the symmetry of the switch design, only one-third of the lateral section was required in order to make the analysis fast and precise (finer mesh elements can be used). Table 4.1 lists the thermal properties of materials, and Table 4.2 lists the contact resistance of the mating surfaces used in the model.

### 4.4.2 Results of the Analysis and Discussion

![Figure 4.2: Temperature profile, side view](image)

For steady state analysis a temperature gradient of 28 K (maximum of 120 K, minimum of 92 K and average of 108 K) was obtained uniformly throughout the heat transfer cross-section (Figures 4.2 and 4.3). A response time of 2.5 hrs was obtained from the transient analysis.
The temperature gradient across the heat conducting section (moving and stationary plates, support rods and bushing) was expected. However, the SMA spring shows a considerable gradient that suggests the stationary plate end of the SMA spring needs to be further isolated thermally. Hence, insulation needed to be provided between the Teflon spring seat and the stationary plate. A response time of 2.50 hrs to reach the steady state is good, considering the methane dewar cool down time of 3.47 hrs (section 4.2).
This chapter discusses the various processes required to fabricate the SMA elements for the thermal switch. Given that the application-specific cryogenic NiTi alloys are not commercially available; the SMA elements for the prototype were decided to be made from commercially available NiTi alloys that have the lowest possible transformation temperatures. Following this, 0.085” (0.216 cm) diameter NiTi wire (56.14 wt. % Ni nominal composition) with an $A_s$ of 234 K was obtained from Special Metals Corp., NY. This wire was used as the starting material for making the SMA helical springs (SMA element).

5.1 **Thermo-Mechanical Processing**

Shape setting the SMA element is a process of setting an original or parent shape that the element will return to upon heating (from the martensitic phase to the austenitic phase). Shape setting is done by constraining the SMA element into the required shape using a fixture or mandrel and then heating it to temperatures of around 673 K to 773 K for 1 to 20 minutes, depending on the size of the element. Typically shape setting is the final thermo-mechanical step. However, in this case due to the starting composition of the wire used, subsequent treatments were performed to enhance the properties.
A cylindrical mandrel was made from 1” diameter brass rod having a helical groove (pitch = 4 turns/inch, length = 3.5”) of roughly 0.085” diameter, with a provision for fastening the wire on one end and an open length on the other end. A lathe was selected for winding the SMA wire on the mandrel. Earlier attempts to wind the wire manually did not work due to the considerable force required and spring back action of the superelastic SMA wire. To begin, the open length of the mandrel was secured to the chuck (Figure 5.1). Subsequently, one end of the SMA wire of suitable length was fixed to the fastening provision on the mandrel and the chuck was hand rotated slowly. A steel sleeve was then pushed over the mandrel (Figure 5.1) as the SMA wire was being wound, hence securing the SMA wire tightly inside the groove. This was continued until the SMA wire was fully wound on the mandrel.
The mandrel-sleeve assembly was then heat treated in a Fisher Isotemp® programmable muffle furnace (Figure 5.2) at 673 K for 20 minutes, followed either by furnace cooling or ice-water quenching.

The SMA wire was tested for its shape memory effect before any kind of heat treatment. It failed to undergo a martensitic transition (poor shape memory), remaining in the austenitic form even when liquid nitrogen temperatures were reached. With the wire set to some form and then annealed above 673 K for 10-20 minutes, the shape memory was restored. This is because the manufacturing process of the SMA wires (wire drawing) resulted in severe deformation (cold working). NiTi when heavily cold-worked introduces high densities of random dislocations that obstruct the mobility of twin boundaries and consequently exhibit poor shape memory [Melton, 1990]. However the yield strength is considerably improved during cold working. Annealing restores shape memory but decreases yield strength.
Table 5.1: Shape setting history

<table>
<thead>
<tr>
<th>Spring Number</th>
<th>Parameters</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spring 1 (1” long)</td>
<td>691 K, 20 min., oven cooled</td>
<td>Very low stiffness compared to the original wire ($A_f$ way above room temperature), and thud sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 2 (1” long)</td>
<td>703 K, 20 min., water quenched</td>
<td>Stiffer than #1 ($A_f$ still above room temperature), and feeble ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 3 (1” long)</td>
<td>678 K, 20 min., ice-water quenched</td>
<td>Stiffer than #1 and 2 ($A_f$ just above room temperature), and good ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 4 (3.5” long)</td>
<td>679 K, 20 min., ice-water quenched</td>
<td>Stiffer than #1 and 2 ($A_f$ just above room temperature), and good ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 5 (3.5” long)</td>
<td>680 K, 20 min., ice-water quenched</td>
<td>Stiffer than #1 and 2 ($A_f$ just above room temperature), and good ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 6 (3.5” long)</td>
<td>679 K, 20 min., ice-water quenched</td>
<td>Stiffer than #1 and 2 ($A_f$ just above room temperature), and good ringing sound when dropped on hard surface.</td>
</tr>
</tbody>
</table>

Table 5.1 highlights the shape setting results of the NiTi springs. Comparison of furnace cooling with ice-water quenching after the heat treatment showed that ice-water quenching made the SMA spring stiffer at room temperature (lowered the $A_f$). This is because during furnace cooling Ni-rich precipitates are formed that deplete the amount of Ni in the matrix, thus elevating the $A_f$. Quenching in ice-water reduces the possibility (time available to form and grow the precipitates) of these Ni-rich precipitates, eventually lowering the $A_f$. However, the method of cooling after heat treatment does not matter if further heat treatments (similar to solution or ageing treatments) are planned.
NiTi alloys are solution treated to improve their properties and tailor transformation temperatures. Such treatments are usually followed by quenching. A subsequent ageing treatment may also be done followed by quenching, without which the transformation temperatures tend to shift during thermal cycling in Ni-rich alloys [Miyazaki et al. 1986]. The solution treatment is generally done at temperatures between 1073 K and 1273 K, and the time depends on the composition of the alloy. Ageing is done at temperatures between 573 K and 973 K for normally 1 hour. Quenching is usually done in oil or water. Inert atmosphere or vacuum heat treatments are necessary to prevent oxidation.

Both the heat treatments were carried out under vacuum conditions in an IVI Corp. vacuum furnace with a quench setup (Figure 5.3). Solution treatment was done at 1073 K for 2 hours (spring#6) followed by quenching in vacuum quenching oil. Ageing treatment was done at 934 K for 1 hour followed by quenching in vacuum quenching oil. The ageing treatment followed by quenching at a high temperature (> 773 K) inhibits the formation of Ni-rich precipitates, which will ensure a lower austenitic transformation range. If aged at 673 K the NiTi
decomposes into a Ti-rich matrix composition with higher austenitic start, together with a Ni-rich phase finely dispersed in it [Melton, 1990]. Furthermore, ageing at higher temperatures suppresses the R-phase transformations and superelasticity in Ni-rich binary alloys.

Table 5.2: Thermal treatment history

<table>
<thead>
<tr>
<th>Spring Number</th>
<th>Parameters</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spring 5 (3.5” long)</td>
<td>Solution treatment at 1177 K, 90 min. and 6.4 x 10^{-5} torr; vacuum-oil quenched</td>
<td>Shape of the spring was severely distorted; excellent ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td>Spring 6 (3.5” long)</td>
<td>Solution treatment at 1073 K, 120 min. and 5.1 x 10^{-5} torr; vacuum-oil quenched</td>
<td>Excellent ringing sound when dropped on hard surface.</td>
</tr>
<tr>
<td></td>
<td>Ageing treatment at 934 K, 60 min. and 5.4 x 10^{-5} torr; vacuum-oil quenched</td>
<td>Discoloration on surface; excellent ringing sound when dropped on hard surface.</td>
</tr>
</tbody>
</table>

For both these heat treatments, the SMA spring was suspended from the work lowering device in the furnace by using a cage made of AISI 300 series stainless steel wires (19 gauge) covered with OMEGA NEXTELF ceramic sleeving. The cage protects the SMA spring from changing its shape during the heat treatment. The ceramic sleeving ensures separation between the stainless steel wire and the NiTi spring; contacts involving them will form a eutectic between Ti and steel at high temperatures, with a possibility of snapping off the steel wire at lower than melting temperatures. Additionally, thin Ti foils were attached to the outer sleeving of the cage to serve as getters and to minimize the oxidation of the NiTi spring.
Table 5.2 highlights the thermal treatment results of NiTi springs. Spring #5 was severely distorted during solution treatment due to lack of proper caging and the relatively high temperatures. Hence, an ageing treatment was not attempted. Spring #6 was successfully solution treated and aged. There was discoloration in the spring, probably as a result of mild oxidation.

5.2 Dilatometric Measurement of Transformation Temperatures

The transformation temperatures and the hysteresis of the SMA spring were measured using a dilatometric setup shown in Figure 5.4. The setup consisted of a helical spring fixture made of Al (Figure 5.5), whose top part moved with the compression or elongation of the SMA spring, contained in an Aluminum cup containing Syltherm XLT® (Cryogenic Heat Transfer Fluid) which in turn was placed inside a small polystyrene (Styrofoam®) tray. A digital dial gauge mounted on top of the helical spring fixture measured the deflection of the SMA spring with temperature, while a thermometer with thermocouple probes measured the temperature of the SMA spring.
The measurement began with a cooling cycle followed by a heating cycle, the cooling being done by pouring liquid nitrogen inside the polystyrene-foam tray gradually. Simultaneous measurements of temperature and deflection were made at regular intervals. The cryogenic heat transfer fluid allows a relatively slow cooling ramp compared to direct cooling with liquid
nitrogen. Temperatures as low as 150 K were attained. The heating was by natural convection until near room temperature, and hot water was used for temperatures above room temperature.

5.2.1 Results and Discussion of Dilatometric Measurements

Dilatometric measurements of transformation temperatures were made on the SMA spring prepared by shape setting before (Figure 5.6) and after (Figure 5.7) the thermal treatments. Table 5.3 shows a comparison of transformation temperatures obtained from both dilatometric measurements. It was observed that subsequent thermal treatments lower the transformation temperatures significantly. Also the hysteresis was reduced by 25 K. The fact that the ageing treatment was done at a high temperature (> 773 K) followed by quenching ensured a lower austenitic transformation range.

Figure 5.6: Graph showing temperature vs. deflection of the SMA spring before the thermal treatment
Figure 5.7: Graph showing temperature vs. deflection of the SMA spring after the thermal treatment.

Table 5.3: Effect of the thermal treatment on transformation temperatures

<table>
<thead>
<tr>
<th>Thermal Treatment</th>
<th>Before</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>$M_s$</td>
<td>273 K (0.0°C)</td>
<td>253 K (-20.0°C)</td>
</tr>
<tr>
<td>$M_f$</td>
<td>213 K (-60.0°C)</td>
<td>193 K (-80.0°C)</td>
</tr>
<tr>
<td>$A_s$</td>
<td>283 K (10.0°C)</td>
<td>237 K (-36.0°C)</td>
</tr>
<tr>
<td>$A_f$</td>
<td>321 K (48.0°C)</td>
<td>285 K (12.0°C)</td>
</tr>
<tr>
<td>$M_p$</td>
<td>~ 243 K (-30.0°C)</td>
<td>~ 213 K (-60.0°C)</td>
</tr>
<tr>
<td>$A_p$</td>
<td>~ 303 K (30.0°C)</td>
<td>~ 248 K (-25.0°C)</td>
</tr>
<tr>
<td>Hysteresis ($A_p - M_p$)</td>
<td>~ 60 K (60°C)</td>
<td>~ 35 K (35°C)</td>
</tr>
</tbody>
</table>
6.1 Fabrication of Prototypes

Based on the final design (model I) discussed in Chapter 3, design drawings of each component were made (see Appendix B for drawings). The components were then sent for fabrication/machining. Three, 1” long SMA helical springs were cut out from the 3.5” long spring after the thermal treatments (Chapter 5). The bias spring was custom fabricated from an outside vendor. Figure 6.1 shows the assembled view of the thermal switch using helical compression springs as SMA elements.
6.2 **Test Setup**

Since the actual working conditions of the switch would be in cryogenic and vacuum conditions, the test setup was expected to simulate those conditions. However any kind of cost-effective setup with extreme vacuum conditions prevents complete monitoring and accessibility to the thermal switch. As it was crucial that complete visibility and accessibility of the thermal switch were required during the technology demonstration phase, a reasonably low-vacuum glove box was considered as an environment for the setup. The cryogenic requirements were achieved through a viable cooling system that makes use of liquid nitrogen.

6.2.1 **Glove Box**

![Glove box with cryogenic setup inside (without insulations)](image)

Figure 6.2: Glove box with cryogenic setup inside (without insulations)
A custom glove box (Figure 6.2) was fabricated with a main chamber having 1” thick clear acrylic (with no tint) on the faces, 1.25” thick on sides, 1.25” wide x 1.5” thick vertical ribs at 10.5” from sides and a 0.5” thick clear acrylic cylinder for the ante-chamber (see Appendix B for drawings). The main chamber has two glove ports in the inclined front side. The glove ports can be used with a pair of gloves when maintaining an inert atmosphere or can be closed using aluminum covers when low vacuum conditions are required. The aluminum covers have o-rings for hermetrical seals and allow the passage of a series of fine thermocouple wires using a feed-through in one of them. The main chamber has a large bolted side door with a Viton gasket (for a hermetrical seal). The ante-chamber has two hinged doors (one on the inside of the main chamber and the other outside) with o-rings (for hermetrical seal), secured by push down clamps. Provisions were made on the main chamber for liquid nitrogen inlets and outlets for the cryogenic setup. The ante-chamber has a brass ball valve for argon inlet and a high vacuum brass ball valve for connecting to a rotary vane high vacuum pump. A glycerin-filled vacuum compound gauge measures the vacuum inside the ante-chamber.

Initially it was thought that the main chamber could take a 25 in. of Hg vacuum pressure considering the thickness of the acrylic sheets used. However simulations using I-DEAS Master Series version 9 showed that it would take a safe vacuum of only 10 in. of Hg vacuum. The vulnerability for buckling was found at the front side of main chamber (Figure 6.3) in between the glove ports, with a possible leakage when pressure is reduced below 15 psi vacuum.
Figure 6.3: I-DEAS simulation of glove box
6.2.2 Cryogenic Setup

![Image of cryogenic setup with text](image)

Figure 6.4: Left - 316 stainless steel tank with Teflon gasket, right - assembled 316 stainless steel tank with lid

The purpose of the cryogenic setup is to simulate feasible cryogenic testing conditions without the use of expensive cryocoolers. For this, two 0.125” thick (0.25” thick on the front side) 316 stainless steel rectangular tanks, having a volume of 8 x 5 x 7 cubic inches (Figure 6.4) were made to replicate the oxygen (hereafter referred to as “Tank-O”) and methane dewars (hereafter referred to as “Tank-M”). They were cooled by copper tubes (with baffle plates) transporting liquid nitrogen, connected to the top lids, with Syltherm XLT® (Cryogenic Heat Transfer Fluid) as the cooling medium inside the tanks. The top lids were bolted to the tanks, with a Teflon gasket in between. The top lids have a feed-through for inserting thermocouple probes in addition to the liquid nitrogen inlet and outlet barbed fittings. Threaded screw holes were made on the front face of Tank-M for mounting the SMA thermal switch (see Appendix B for drawings). During the trial run it was found that copper tubes with a number of loops were more efficient and have a better rate of cooling than the single loop with baffle plates (even...
though the final temperatures reached remains the same). For this reason, the single loop copper tube with baffle plates (Figure 6.5, left) in the Tank-O was replaced with one with a number of loops (Figure 6.5, right).

![Figure 6.5:Left - lid connected to single loop copper tubes with baffle plates, right - lid connected to copper tubes with a number of loops](image)

Liquid nitrogen from the low-pressure liquid nitrogen cylinder was passed through a brass pressure regulating valve (Figure 6.6). It then diverged through a brass tee, with brass flow regulating valves on either end. The other ends of the regulating valves were connected to the inlets of individual tanks, Tank-O and Tank-M, with Teflon tubes. The Teflon tube outlets from each tank merged before being exhausted to the fume-hood.
6.3 Testing Procedures

For the testing of the prototype two approaches were employed: (i) outside chamber actuation, and (ii) inside chamber actuation. In outside chamber actuation, the switch was tested without the glove box or cryogenic setup, where the switch was initially cooled with liquid nitrogen and was allowed to warm up either by natural convection or forced convection (using a heat gun). The actuation (deflection) of the switch was measured with and without load. In inside chamber actuation, the switch was tested by mounting it on the cryogenic setup and then placing the whole assembly inside the glove-box. The atmosphere was low vacuum or argon atmospheres to prevent or reduce the formation of frost.

6.3.1 Outside Chamber Actuation

Initially the SMA elements were cooled down to liquid nitrogen temperatures by slowly pouring liquid nitrogen over the elements. The switch was then placed over a flat surface without
any external load, with a digital dial gauge to measure the deflection. It was then allowed to warm up either using natural convection or forced convection (using a heat gun). The deflection was measured and the actuation was filmed (forced convection). Another trial under natural convection with a set of deflection versus temperature (with thermocouple attached) readings were taken. This procedure was then repeated with an external load of 20 N (68 oz.) by using a container of lead shots for loading.

### 6.3.2 Inside Chamber Actuation

For testing the SMA thermal switch by inside chamber actuation, the thermal switch was mounted on the Tank-M with an indium foil in between. A copper disc covered with indium foil (for thermal contact) was mounted on Tank-O. Type K thermocouple probes were inserted into the feed-through provided on the top of each tank. The two rectangular tanks, insulated by 0.75” thick polystyrene-foam (Styrofoam®) sheets, were then placed on a 0.25” thick Teflon sheet with adjustable, L-shaped angle plates mounted on it (see Appendix B for drawings). The L-shaped angle plates help keep the SMA thermal switch (connected to Tank-M) in thermal contact with the Tank-O, with proper contact force. The whole assembly was placed inside the glove box through the side opening, on a 0.75” thick polystyrene-foam insulation base. Two to three fine type-T thermocouple wires were attached to the SMA springs at various locations to get the temperature reading. These type-T thermocouple wires came out of the glove-box through a feed through attached to the aluminum covers of the glove ports. They were connected to a multiple input handheld digital thermocouple thermometer. The thermocouple probes from individual
tanks were connected to a dual input handheld digital thermocouple thermometer which was kept inside the glove box (Figure 6.7).

Figure 6.7: Setup for inside chamber actuation

Figure 6.8: Photo showing Teflon tubing for liquid nitrogen inlet and outlet connections to the tanks
The liquid nitrogen inlet and outlet Teflon tubing connections passed through tank-adapters (316 stainless steel) fitted to the glove-box (Figure 6.7). The in-house fabricated Teflon seal bushings (see Appendix B for drawings) ensured a hermetical seal. The Teflon tubing connections were then connected to the barbed fittings on individual tanks (Tank-O and Tank-M) and secured with 316 stainless steel worm-drive hose clamps (Figure 6.8). The side opening was then bolted with the side door and the two glove ports were covered with the aluminum covers.

After completing the setup procedures discussed above, each test run started with passing Ar gas at a pressure of 10 psi (gage) through the glove box. This reduced the amount of water vapor present inside the glove box. The argon flow was stopped after 2 to 3hrs. Initial thermocouple readings of the tanks and those at various points on the switch were taken. Initially, the flow regulating valve of Tank-M was completely closed and that of Tank-O was fully opened. Subsequently, the flow of liquid nitrogen was started, with the flow valve of the liquid nitrogen dewar set for low flow. The thermocouple readings were taken every 15 minutes and any change in conditions (either observed or forced) was recorded. This procedure was continued until an hour after actuation was observed.
CHAPTER SEVEN: PROTOTYPE TESTING RESULTS, DISCUSSIONS AND RECOMMENDATIONS

7.1 Prototype Testing Results

The prototype after assembly was thermally cycled more than 10 times to (i) train the SMA elements to act together and (ii) to reduce any variation of transformation temperatures with cycling, if any. The prototype was then tested for both outside chamber actuation and inside chamber actuation. The results are summarized below.

7.1.1 Outside Chamber Actuation

The outside chamber actuation was measured with and without external load, under forced and natural convection. The actuation for both cases was filmed under forced convection (see Appendix C for movie details), and a set of readings of temperature vs. deflection were taken under natural convection.

An actuation (deflection) of 6.543 mm was measured without external load under forced convection. Here the only load (internal) on the SMA elements came from the bias spring, where the load varies with deflection. An actuation (deflection) of 3.610 mm was measured with a load of 20 N (68 oz.) under forced convection. Here the total load on the SMA elements came from the bias spring (varies with deflection) and the external load (constant). Figure 7.1 shows movie clips of outside chamber actuation without any external load under forced convection and Figure
7.2 shows movie clips of outside chamber action with an external load of 20 N under forced convection.

Figure 7.1: Clips (a to f) showing sequence of action during outside chamber actuation without external load
Figure 7.2: Clips (a to f) showing sequence of action during outside chamber actuation with an external load of 20 N
Figure 7.3 shows deflection as a function of temperature in outside chamber actuation with and without an external load of 20 N under natural convection. There is a difference of 7 K for the onset of austenitic transformation.

Figure 7.3: Graph showing deflection as a function of temperature in outside chamber actuation with and without external load.
7.1.2 Inside Chamber Actuation

The minimum temperature achievable in Tank-O (isolated condition) was found to be 123 K (-150 °C). Initial test results with Tank-O being below 173 K and Tank-M around 233 K to 203 K (ideal test conditions) showed unsatisfactory actuation of the thermal conduction switch. The temperature readings of thermocouples showed making and breaking of thermal contact in an irregular pattern.

However, later trials with both Tank-O and Tank-M cooled down to below 173 K showed sufficient actuation (visual break in contact). Figure 7.4 shows the thermal switch before and during the observed actuation. About 3 mm deflection was observed in this case. Figure 7.5 shows the thermocouple readings as a function of time. Instances of change in conditions (either forced or observed) are denoted by points A to F. At point A, the testing was started with the regulating valve for Tank-O fully open and that of Tank-M completely closed. In between points A and B the Tank-M was being cooled by the Tank-O. At point B the regulating valve of Tank-
M was slightly opened to expedite the cooling of Tank-M. At point C, the regulating valve for Tank-M was closed. In between points C and D the whole system attained a steady state where further cooling of Tank-M is very slow. At point D the regulating valve for Tank-M was half opened. At point E a visible actuation (Figure 7.4, Right) was observed. At point F the regulating valve for Tank-M was closed. Beyond point F the temperatures of Tank-M and SMA springs rises due to the disconnection of thermal contact with Tank-O and the supply of liquid nitrogen to Tank-M being shut-off.

Figure 7.5: Graph showing thermocouple readings of inside chamber actuation as a function of time (SMA temperature near moving plate side)
7.2 **Discussion**

The SMA switch was able to take an external load of 20 N for a difference in deflection of around 2.9 mm during outside chamber actuation. A load of 20 N is sufficient to keep a good thermal contact among copper conductors with indium foil in between, even at liquid helium temperatures [Salerno & Kittel 1998]. The temperature difference of 7 K for the onset of austenitic transformation was expected, as the presence of additional stress (in addition to stress from the bias spring) delays the transformation of martensite to austenite. A difference in deflection of 2.93 mm was observed during the outside chamber actuation with an external load of 20 N. This when extrapolated for a deflection of 5 mm would give an external load of 34.13 N. The value of 34.13 N external load for 5 mm deflection has an excellent agreement with the value of 36 N external load projected from Figure 3.6, addressed in section 3.5.4.

The initial trials of inside chamber actuation with Tank-O below 173 K and Tank-M around 233 K to 203 K showed unsatisfactory actuation. This is due to the fact that a large temperature gradient was developed within the SMA spring. Even when sufficient actuation was obtained during later trials of cooling both tanks below 173 K, this temperature gradient was evident from the SMA spring (Figure 7.4, Right). There was a gradual variation of gap between loops in the SMA spring; the closest being near the moving plate side and the farthest near the opposite end. When such a thermal gradient develops, the transformation to martensite may not be uniform, resulting in fully transformed martensite at the moving plate end and more untransformed austenite at the other end.

The heat transfer rate was calculated for the SMA thermal conduction switch, by measuring the heat rejected by the Tank-M during a time period. The total mass of heat transfer
fluid (Syltherm XLT®) in the Tank-M was calculated to be 3.343 kg from a tank volume of 3.93 \( \times 10^{-3} \) m\(^3\) and density of 850 kg/m\(^3\). The specific heat of the fluid is 1.339 kJ/kg-K (from the manufacturer). During the time when Tank-M was being cooled by Tank-O, the heat transfer rate was 11.937 W for a temperature difference of 52 K between Tank-O and Tank-M. This would mean a heat transfer rate of around 6.5 W for a temperature difference of 28 K that is comparable to the required 8 W. Additionally, when the factors of losses due to convection were taken into account, the heat transfer rate would get nearer to 8 W.

7.3 **Solutions to Issues Encountered**

1) Seizure of sliding contacts (bushing and support rods) was encountered at cryogenic temperatures due to freezing of Apiezon® grease into a solid mass. Later another synthetic lubricant (SL-C from SENTINEL®) that is used near cryogenic temperatures was tried. However, it failed to keep the lubricating properties at lower cryogenic temperatures. Later trials with a mixture of Syltherm XLT® (cryogenic heat transfer fluid) along with a small amount of synthetic SL-C (they were mixed at slightly elevated temperatures from room temperature) showed better results.

2) The lowest temperature achievable in the tanks with the cryogenic cooling setup was above 173 K. Covering the tanks with proper insulation helped in achieving temperatures below 173 K, in addition to making them cool faster and more efficient.

3) A temperature gradient developed across the SMA spring, even with the Teflon spring seats. To some extent the gradient was reduced by placing disc-shaped polystyrene insulation in between the Teflon seat and the stationary plate.
4) Unsatisfactory actuation of the first prototype was encountered due to misalignment or difference in thermal expansion of moving parts at cryogenic temperatures. Isolating the bushing and the support rods, and testing them with liquid nitrogen temperatures confirmed that there was no seizure between them. This eliminated the possibility that the problem was due to variable thermal expansion between bushings and support rods. Later, another prototype made from a machining facility having precision capability actuated satisfactorily.

7.4 Recommendations

1) The martensitic hysteresis in SMAs is a major problem in designing an actuator that is supposed to work within a limited temperature range. This problem can be addressed in three ways: (a) Use of R-phase transformation (between austenitic phase and R-phase) that is coupled with a temperature hysteresis as low as 1.5 K. However strains are limited to less than 1%; but have higher fatigue life than martensitic transformation. (b) A combination of thermo-mechanical treatments on a suitable NiTi alloy (tailoring the composition by the addition of a third element). (c) Use of single crystal and single variant SMA elements that do not exhibit transformation hysteresis. However the research in creating single crystal, single variant SMAs is limited and its application has not been utilized.

2) It is better to sense the temperature fluctuations within the methane dewar rather than depend on the fluctuation during the heat transfer that takes place between the oxygen and methane dewars.
3) Keeping the SMA element parallel to conduction path (heat transfer path) will create a temperature gradient within the element (whatever the counter measures are). One way to avoid this may be to design the actuator in such a way to minimize the gradient. A patent is being pursued with this approach in mind.

4) When determining the bias force (use of graph showing austenitic, martensitic/R-phase and bias force vs. deflection), it is better to find the austenitic force at the required maximum temperature (not at room temperature) and the martensitic (or R-phase) force at the required minimum temperature (not at liquid nitrogen temperatures). This can be accomplished by measuring deflection versus temperature under constant load for different load conditions and then plotting load versus deflection for the required temperatures from that [Ohkata & Suzuki 1998]. Such a setup would eliminate any variations in either austenitic or martensitic force with temperature that occur in practice.

7.5 Extension to NASA Requirements

Taking into account the recommendations suggested in section 7.4, suggestions can be made by extending the present design of the switch to meet NASA’s requirements for the SMA thermal conduction switch. The R-phase transformation can be used for designing the SMA thermal conduction switch instead of the martensitic transformation to reduce the transformation hysteresis. Alternatively, additional thermo-mechanical processing steps performed on a suitable NiTi alloy (by tailoring the composition with the addition of a third element) may result in reduced hysteresis within the martensitic transformation. This application specific shape memory alloy would be developed as a part of an ongoing NASA project for the development of
cryogenic shape memory alloys. The use of thin strips rather than helical springs as the SMA elements in the switch would help the make the SMA elements simple to fabricate. The thin strips are made by cutting SMA alloy buttons (see Figure 7.6) processed as a part of the above mentioned project using either an EDM or a slow-speed cutting saw. An SMA thermal conduction switch design (model II) based on thin strips as SMA elements is shown in Figure 7.7. The testing of a prototype (Figure 7.8) based on this design (model II) is currently being pursued. A patent will be filed on the design of SMA thermal conduction as suggested in the recommendation iii (section 7.4), in addition to those discussed (models I and II) in this work.

Figure 7.6: Processing of Ni-Ti-Fe alloys; left - arc melting of Ni-Ti-Fe powders, center - buttons after melting, and right - thermo-mechanically processed SMA strip [Krishnan et al. 2004]
Figure 7.7: Design model II

Figure 7.8: Prototype based on design model II
CHAPTER EIGHT: CONCLUSION AND FUTURE APPLICATIONS

8.1 Conclusion

The design and development of a shape memory alloy (SMA) based cryogenic thermal conduction switch for operation between dewars of liquid methane and liquid oxygen in a common bulkhead arrangement has been presented. Such a switch integrates the sensor element and the actuator element and can be used to create a variable thermal sink to other cryogenic tanks for liquefaction, densification, and zero boil-off systems in advanced spaceport applications. The design of this thermal conduction switch is based on a biased, two-way SMA actuator. This work describes the design of a cryogenic SMA thermal conduction switch from the concept, addressing challenges, design issues and methodologies. A finite element based thermal analysis was carried out to predict the behavior of the thermal conduction switch. SMA springs were made from a commercially available NiTi SMA wire that had the lowest transformation temperatures and various heat treatment processes were carried out on them to enhance their performance. Based on design drawings, an SMA thermal conduction switch using SMA helical springs was effectively fabricated. A testing setup comprised of a glove box and cryogenic setup was constructed. The SMA thermal conduction switch was tested at cryogenic conditions using the testing setup. Successful actuation was achieved within the limitations of the commercially available cryogenic SMA material and conditions. The experience from this work resulted in recommendations for future improvements in the use of SMA materials and design. This work has been able to successfully demonstrate the feasibility of an SMA thermal
conduction switch at cryogenic temperatures. A patent will be filed on the basis of knowledge gained from this project with appropriate modifications.

8.2 Future Applications and Possibilities

The initial intended application of the SMA thermal switch is to provide cooling to cryogenic storage and distribution systems, for liquefaction, boil-off control, and subcooling purposes. However, other future cryogenic applications of SMA thermal switches can range from cooling the sensors of space systems and medical devices, to laboratory applications (adiabatic-demagnetization refrigerators, calorimeters, high performance infrared cameras) where temperature control of materials or systems is required. This switch can also be used to control temperatures of other low-temperature systems that are not in the cryogenic range, such as commercial refrigeration for food, personal cooling suits, and electronics and lasers. Further, it may find applications in maintaining the temperature of future high-temperature superconducting power transmission lines. In addition a reconfiguration of the SMA thermal switch would allow the reverse to take place, that is heating of a low-temperature device by a relatively high temperature source and could be used to maintain temperatures in an industrial or laboratory system [Notardonato W. U., 2002-03]. Another interesting option is in the micro/nano spacecraft thermal control, where MEMS-based tiny SMA thermal switches can be used.

The low-temperature infrared sensors used in space systems need at least two cryocoolers because of their reliability concerns. Of the two cryocoolers, one is a primary (normally ON) and another back-up (normally OFF). A considerable amount of input power is required by these cryocoolers just to produce a small amount of cooling. This is due to the additional burden of the
load coming from the heat that is being transferred from the backup cryocooler. This problem can be solved by using two cryogenic thermal switches in parallel (one for each cryocooler), thus minimizing the flow of heat from the backup cryocooler and maximizing the cooling capability of the primary cryocooler. In case the primary cryocooler fails, the cryogenic thermal switch of the primary can be turned OFF, while the backup cryocooler along with its cryogenic thermal switch can be turned ON [NASA facts, September 1998].
APPENDIX A: DESIGN CALCULATIONS
**SMA Spring Design (3 springs)**

Load on each SMA spring, $P = 16$ N, compressive  (Design Load)

Deflection, $\delta = 10$ mm

Spring index, $C = 12$

**Mechanical Properties for NiTi Spring Material**

$\tau_{\text{max}} = 120$ MPa

$G_L = 2700$ MPa

$G_H = 22000$ MPa

**Calculations**

Stress concentration factor, $K = (4C-1) / (4C-4) + 0.615 / C = 1.1194$

Shear stress, $\tau = \frac{8\ P\ D\ K}{\pi d^3} = \frac{8\ P\ C\ K}{\pi d^2}$

$d^2 = 4.5609$ mm$^2$

$d = 2.1356$ mm

i.e., taking standard size; Wire diameter, $d = 2.1590$ mm (0.085”)

Mean coil diameter, $D = 25.9080$ mm (1.02 “)

$\gamma_H = \frac{\tau}{G_H} = 0.0055$

$\gamma_L = \frac{\tau}{G_L} = 0.0444$ (>1%)

$\gamma_S = \gamma_{\text{max}} - \gamma_H = 0.0045$ [Taking, $\gamma_{\text{max}} = .01$]

Shear strain, $\gamma = \frac{\delta d}{\pi n D^2}$

Number of turns, $n = \frac{\delta d}{\pi \gamma_S D^2} = 2.2525$

Or, $n = 2$

For squared and ground ends, the total number of turns, $n' = n + 2 = 4$

Free length of spring = $n' d + \delta + 0.5 \delta = 23.6360$ mm (0.9305”)
Pitch of the coil = Free Length / (n' - 1) = 7.8787 mm (0.3102”)

Threads per inch = 3.2239

Proposed load = 10 N

High temperature spring rate, $K_H = 1.7180$ N/mm

Low temperature spring rate, $K_L = 0.2108$ N/mm

High temperature deflection, $\delta_H = 5.8209$ mm (0.22917”)

Low temperature deflection, $\delta_L = 15.8209$ mm (0.6229”)

Minimum reset force = $\delta_L \times K_L = 3.3357$ N

1/3 Biasing rate = (high temperature bias force – reset force) / deflection

= $(10 \text{ N} - 3.33357 \text{ N}) / 10 \text{ mm} = 0.6664 \text{ N/mm}$

Biasing rate, $K_{Bias} = 1.9993$ N/mm

Bia spring load = 19.9930 N

(Note: - the values of $K_H$ and $K_L$ were arrived by testing the SMA spring for deflection vs. load after heat treatments)

**Bias Spring Design (1 spring)**

Load on bias spring, $P = 20$ N, tensile

Deflection, $\delta = 8$ mm

Spring index, $C = 10$

**Mechanical Properties**

Material for spring:- stainless steel AISI 302 (austenitic)

$\tau_{\text{max}} = 227.7$ MPa

$G = 69000$ MPa
Calculations

Stress concentration factor, \( K = \frac{4C-1}{4C-4} + 0.615/C = 1.1448 \)

Shear stress, \( \tau = \frac{8P DK}{\pi d^3} = \frac{8PCK}{\pi d^2} \)

\( d^2 = 2.5606 \text{ mm}^2 \)

\( d = 1.6002 \text{ mm} \)

i.e. taking standard size (AWG 14); Wire diameter, \( d = 1.6280 \text{ mm (0.0641”)} \)

Mean coil diameter, \( D = 16.28 \text{ mm (0.6409”)} \)

Load, \( P = \frac{\delta G d^4}{8 n D^3} = \frac{\delta G d}{8 n C^3} \)

Number of turns, \( n = \frac{\delta G d}{8 P C^3} = 5.6166 \)

Or, \( n = 6 \)

Free length of spring = \( n d = 9.768 \text{ mm (0.3846”)} \)

Pitch of the coil = Free length / (\( n - 1 \)) = 1.9536 mm (0.0769”)

Heat Transfer Area Required when Using Copper Medium

Required heat rate, \( Q' = 8 \text{ W} \)

Minimum temperature difference, \( T_1 - T_2 = 118 \text{ K} - 92 \text{ K} = 26 \text{ K} \)

Assuming stainless steel dewars for oxygen and methane

\( K_{304ss} = 11.17 \text{ W/m-K (at 120K)} = K_1 \)

\( K_{304ss} = 9.592 \text{ W/m-K (at 92K)} = K_2 \)

\( K_{Cu} = 401 \text{ W/m-K} \)

Assuming thickness to be, \( x_1 = x_2 = 0.003175 \text{ m} \)

Assuming the length in between oxygen and methane dewars, \( L = 0.05 \text{ m} \)

\( Q' = \frac{(T_1 - T_2)}{[x_1 / K_1. A] + (L / K_{Cu}. A) + (x_2 / K_2. A)]} \)
\[ A = \left( \frac{Q'}{(T_1 - T_2)} \right) \left[ \left( \frac{x_1}{K_1} \right) + \left( \frac{L}{K_{Cu}} \right) + \left( \frac{x_2}{K_2} \right) \right] \]

\[ = 0.0002277 \text{ m}^2 \]

\[ = 227.6729 \text{ mm}^2 \]

Number of straps req. (0.508mm thick x 50.8mm wide) \[ = 8.8223 \]

Required thickness of F-shape conductor having width 12.7mm (0.5") \[ = 7.9375 \text{ mm (0.3125")} \]

**Heat Transfer Area Required when Using BeCu Medium**

\[ K_{BeCu} = 41.4085 \text{ W} / \text{m-K (at 100 K)} \]

Effective length of beryllium-copper rod, \( L = 0.0254 \text{ m} \)

\[ A = \left( \frac{Q'}{(T_1 - T_2)} \right) \left[ \left( \frac{x_1}{K_1} \right) + \left( \frac{L}{K_{BeCu}} \right) + \left( \frac{x_2}{K_2} \right) \right] \]

\[ = 0.000378 \text{ m}^2 \]

\[ = 378.0458904 \text{ mm}^2 \]

Required diameter when using 3 support rods = 12.6668  \text{ mm (0.4987")} 

**Force vs. Deflection for the Bias Spring and SMA Spring in Austenitic and Martensitic Conditions**

Austenitic force = \( K_H \times \text{deflection} \)

Martensitic force = \( K_L \times \text{deflection} \)

Bias force = \( [\text{deflection} \times 1/3 \ K_{Bias}] + [\delta_H \ (K_H + 1/3 \ K_{Bias})] \)
<table>
<thead>
<tr>
<th>Defection (mm)</th>
<th>Austenitic Force (N)</th>
<th>Martensitic Force (N)</th>
<th>Bias Force (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.718</td>
<td>0.210</td>
<td>13.213</td>
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<tr>
<td>2</td>
<td>3.436</td>
<td>0.420</td>
<td>12.546</td>
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<td>0.630</td>
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<td>10.308</td>
<td>1.260</td>
<td>9.881</td>
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<td>7</td>
<td>12.026</td>
<td>1.470</td>
<td>9.214</td>
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<td>13.744</td>
<td>1.680</td>
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<td>10</td>
<td>17.180</td>
<td>2.100</td>
<td>7.215</td>
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<tr>
<td>11</td>
<td>18.898</td>
<td>2.310</td>
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<td>36.078</td>
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</tbody>
</table>

**Heat Transfer Requirements for Oxygen and Methane Dewars**

Specific heat of methane at 118 K, $C_p = 3530$ J/K·kg

Density of methane = 424 kg / m³

Capacity of dewars = 25 L = 0.025 m³

Temperature difference = 120 K – 118 K = 2 K

Quantity of heat, $Q = 74836$ J

Time required to raise temperature due to heat leakage (@ 2W) = 37418 s

= 10.3939 hrs.

Time required bringing back methane tank from 120K to 118K (@ 8W)

= 12472.6667 s

= 3.4646 hrs. (6 W Effective)
APPENDIX B: DESIGN DRAWINGS
Base Plate - Tank side

AMPAC

Note:
Material - Oxygen free Copper
Note:
Material - Oxygen free Copper

AMPAC

Base Plate - Moving

All Dimensions in Inches

Designed & Drawn By: Vina Krishna
Note:
Material: Brass
2 pieces required

Bias-Spring Holder

AMPAC

First Angle Projection  DWG NO. 123  Sheet 1/1  Rev. 0

All Dimensions in Inches  Designed & Drawn By: Vimla Krishnan
Circular Groove 0.125" deep

Section X'-X'

Note:
Material: Teflon
3 pieces required

AMPAC

SMA-Spring Seat

All Dimensions in Inches

Designed & Drawn By: Vinu Krishnan
Note:
Material: Beryllium Copper C17200, HT (TH04)
Straightness Required: ±0.0005 in./in.

AMPAC
Support Rod

First Angle Projection

All Dimensions in Inches

Designed & Drawn By: Vina Krishna
Note:
Material: Beryllium Copper C17510, TF00

AMPAC

Bushing

AllDimensions in Inches  Designed & Drawn By: Vina Krishna
Note:
Material - Oxygen free Copper

AMPAC

Base Plate (m-2)-Tank side

First Angle Projection  DWG NO. 127  Sheet 1/1  Rev. 0

All Dimensions in Inches  Designed & Drawn By: Vam Krishnam
Note:
Material - Oxygen free Copper

AMPAC
Base Plate (m-2) - Moving

@ First Angle Projection  DWG NO. 128  Sheet 1/1  Rev. 0

All Dimensions in Inches  Designed & Drawn By: Vinit Krishna
Note:
Material: Copper
15 pieces required
Note:
Material: Clear acrylic with no tint
Edges (Outside): 1.5" wide x 1.25" thick
Edges (Inside): 1.5" wide x 1.25" thick
Top, bottom, front & back faces: 0.0625" thick
End faces (Main Chamber): 1.25" thick
For other dimensions see drawings 2/35, 3/35, 4/35 & 5/35

AMPAC
Vacuum Glove Box Assembly
Note:
This drawing shows main body without doors.
Material: Clear acrylic with no tint
Eibs (outside) - 1.5" wide x 1.25" thick
Eibs (inside) - 1.5" wide x 1.25" thick
Top, bottom, front & back faces - 1.0" thick
Induced (main chamber) - 1.25" thick
For detail dimensions see drawings 3/5, 4/5 & 5/5

AMPAC
Vacuum Glove Box - Main Body
Main Chamber Door

Circular Groove for O-Ring
(1 1/2" O.D., 3/16" H & 1/8" Deep)

Ante-Chamber Door
(2 Numbers required)

Note:
Material: Clear acrylik with no tint
One Ante-Chamber Door is for inside Main Chamber
(connecting Ante-Chamber)
The other door covers the outside of Ante-Chamber
The O-Ring Groove is Hemispherical in shape

AMPAC
Vacuum Glove Box - Doors

First Angle Projection

Designed & Drawn By: Venu Krishna
Note:
Material: 316 Stainless Steel
Weld using 316 L welding rod
Front face 0.25" thick Mirror finish
All other faces and bottom face 0.125" thick
Flange 1" wide x 0.187" thick
Note:
Material - 316 Stainless Steel
Weld using 316 L welding rod
Front face 0.25" thick Mirror finish
All other faces and bottom face 0.125" thick
Flange 1" wide x 0.187" thick

AMPAC

316 Stainless Steel Tank 2 (8 x 5 x 7 Cu. in.)
AMPAC

316 Stainless Steel Tank Top Cover

Note:
Material - 316 Stainless Steel
2 pieces required
Note:
Material - Teflon
Provide thread relief
3 pieces required

AMPAC
Seal Bushing

First Angle Projection
D WG NO. 191
Sheet 1/1
Rev 0

All Dimensions in Inches
Designed & Drawn By: Viru Krishna
APPENDIX C: OUTSIDE CHAMBER ACTUATION MOVIES
Two movies named Krishnan_Vinu_B_200405_MS_sma switch without load.avi and Krishnan_Vinu_B_200405_MS_sma switch with load.avi are included as a supplementary material. The content of these two movies are discussed below.

**SMA Switch without Load**

The outside chamber actuation was filmed without any external load under forced convection. Here the only force acting against the SMA spring is that of the bias spring. Initially the SMA elements were cooled down to liquid nitrogen temperatures by slowly pouring liquid nitrogen over the elements. The switch was then placed over a flat surface, with the digital dial gauge to measure the deflection. It was then allowed to warm up by forced convection (using a heat gun). A deflection of 6.543 mm was measured.

**SMA Switch with Load**

The outside chamber actuation was filmed with an external load of 20 N under forced convection. Here the forces acting against the SMA spring is that of the bias spring and the external force of 20 N. Initially the SMA elements were cooled down to liquid nitrogen temperatures by slowly pouring liquid nitrogen over the elements. The switch was then placed over a flat surface with an external load of 20 N (by using a container of lead shots weighing 68 oz. for loading), with the digital dial gauge to measure the deflection. It was then allowed to warm up by forced convection (using a heat gun). A deflection of 3.610 mm was measured.
REFERENCES


