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Shape Memory Alloy Actuator and Fluid Sampler Development for Extreme Environments

Rachel Stolzman

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Santa Clara University

Department of M echanical Engineering

Date: June 20th, 2020

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Rachel Stolzman

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Shape Memory Alloy Actuator and Fluid Sampler Development **for Extreme Environments**

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Shape Memory Alloy Actuator and Fluid Sampler Development for Extreme Environments

By

Rachel Stolzman

Graduate Thesis

Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science in Mechanical Engineering in the School of Engineering at Santa Clara University, 2020

Santa Clara, California

Shape Memory Alloy Actuator and Fluid Sampler Development for Extreme Environments

Rachel Stolzman

Department of Mechanical Engineering Santa Clara University Santa Clara, California 2020

ABSTRACT

Due to the high temperature, high pressure environments of deep-sea boreholes, using conventional electronics to capture temperature-specific water samples may be unreliable or even impossible. In this project, we introduce the use of shape memory alloys as thermal sensors to trigger an actuator to take a water sample at a specified temperature. A custom sampler that can interface with a variety of developed triggers was also designed and fabricated for use in deep-sea boreholes. All of the developed triggers are modular and designed for ease-of-use by users on a drillship with limited tools and equipment. The shape memory alloys used in these triggers vary in shape and activation temperatures, ranging from 80°C to 170°C. This thesis presents the development of the sampler as well as several of the trigger designs. Two of the trigger designs utilizing four different shape memory alloys were fabricated and tested in-house before deployment on IODP Expedition 385T.

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Due to the high temperature, high pressure environments of deep-sea boreholes, using conventional electronics to capture temperature-specific water samples may be unreliable or even impossible. In this project, we introduce the use of shape memory alloys as thermal sensors to trigger an actuator to take a water sample at a specified temperature. A custom sampler that can interface with a variety of developed triggers was also designed and fabricated for use in deep-sea boreholes. All of the developed triggers are modular and designed for ease-of-use by users on a drillship with limited tools and equipment. The shape memory alloys used in these triggers vary in shape and activation temperatures, ranging from 80°C to 170°C. This thesis presents the development of the sampler as well as several of the trigger designs. Two of the trigger designs utilizing four different shape memory alloys were fabricated and tested in-house before deployment on IODP Expedition 385T.

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Nomenclature

Chapter 1 Introduction

The sampler and corresponding triggers outlined in this thesis were initially developed for travel to boreholes 896A and 504B in the summer of 2019 on IODP Expedition 385T. Dr C. Geoffrey Wheat of the University of Alaska, Fairbanks in collaboration with Dr Christopher Kitts of Santa Clara University were awarded a National Science Foundation grant to fund the development of this sampler and various triggers for the purpose of exploring the thermal limits of life forms. Development of the sampler and several triggers spanned the course of nine months before deployment, and then a gastight design for future iterations was drafted during the second year.

1.1 Deep-Sea Boreholes

In the 1940's, advancements in piston coring finally allowed for research ships to take samples of the earth's crust in oceans around the world. Deep-sea boreholes can reach depths as low as five kilometers below the seafloor and water temperatures up to 200°C. Due to immense pressures, the overheated water is able to stay in a liquid state. The first promising project in deep-sea drilling, Project Mohole, was proposed in 1961 by the US National Science Foundation. The Ocean Drilling Program then began in 1983 as a joint venture with various countries to conduct expeditions to drill holes around the world. Starting in 2003, the ODP expanded with more drillships and country participation to form the Integrated Ocean Drilling Program. In 2013, IODP changed their name to the International Ocean Discovery Program to better reflect the objective of researching over drilling [1].

Deep-sea boreholes around the world help scientists better understand the composition of the Earth as well as the nature of micro-organisms in extreme environments. These

boreholes are largely left undisturbed for years, creating an ideal environment to sample and observe. Different geographical locations and depths will provide different temperature gradients and, in some cases, different compositions of fluid. The structure of a borehole begins at the bottom of the seafloor with a concrete-lined hole of about 12 inches in diameter. To access the borehole, a drillship finds this 12-inch diameter hole and hovers over it, lining up the drillship assembly gear to travel down the hole. From the 12 inches, the hole tapers inward to about 4.25 inches, and can have sections as narrow as 3.75 inches. This largely determines the dimensions of the gear that can be sent down a borehole. At the bottom end of the drillship assembly is a drilling bit to move dirt and other sediment out of the way of the assembly [2].

Figure 1.1: Diagram of drillship with assembly lowered into a cartoon version of a deep sea borehole (used without permission) [3].

Hole 504B is one of the largest in the world, located about 200 km south of the Costa Rica Rift. 504B reaches depths of 3.46 km and recorded temperatures of up to 150°C. Hole 896A is also located near the Costa Rica Rift. This borehole is a shallower, lower temperature borehole than 504B with less recorded information of the microbial content $|5|$.

1.2 Shape Memory Alloys

Shape memory alloys (SMA's) are metallic alloys that deform by phase transformation due to temperature. They can "remember" a predetermined shape after plastic deformation when heated to its activation temperature. This return to a predetermined shape is called shape memory effect (SME) [6]. These alloys are especially useful when conventional electronic sensors are not able to perform to their expected tolerance.

1.2.1 Shape Memory Effect

The phase transformation of an SMA occurs when the alloy changes between its Austenite phase and Martensite phase. There are two Martensitic phases with different crystal structures, referred to as twinned Martensite and de-twinned Martensite. The SMA in its moldable form is in its Martensitic phase whereas the SMA in its "remembered" shape is in its Austenitic phase [7].

Figure 1.2: Phase transformation graph demonstrating effect of temperature (used without permission) [7].

As the material cools down from its Austenitic phase, it enters its twinned Martensitic phase, meaning the shape will look the same if left undisturbed but the SMA can now be molded into any shape that the mechanical properties allow for. Once the SMA is deformed, it is now in its de-twinned Martensitic phase and remains there until heated to its activation temperature. At the activation temperature, the SMA returns to its Austenitic phase [6].

Figure 1.3: Heating and cooling curve demonstrating hysteresis of SMAs (used without permission) [7].

Overheating an SMA will alter its properties and can potentially cause the SMA to lose its shape memory effect. To change the remembered shape, the SMA must be mechanically fixed into its new shape, either with a mold or clamped into position, and annealed at high temperatures for a few hours. Heating at too high of temperatures will cause the SMA to lose shape memory properties [6].

There is a possible "two-way" SMA that has both a high and low temperature configuration, meaning at low temperatures instead of needing a force to deform it into a certain shape, it automatically returns back to its pre-determined cold shape. This two-way shape memory effect is created through several processes done on the material. The disadvantages of two-way SMAs are limited range of transformation strain, instability of material, and unpredictable repeatability [5].

Figure 1.4: An example of a 2-way SMA with its heated state on the left and cooled state on the right (used without permission) [8].

1.2.2 M aterial Properties

SMA's can exist in a variety of configurations, shapes, and material compositions. The most common SMA commercially available are nickel-titanium blends with actuation temperatures typically between 30°C and 130°C. For higher temperatures, blends such as copper-aluminum-nickel (CuAlNi) and nickel-titanium-copper (NiTiCu) can be used. Tweaking the composition ratios will also change the activation temperatures. For example, the introduction of copper in NiTi SMAs affects not just the activation

temperature but will also narrow the hysteresis gap. Palladium to NiTi SMAs can increase the activation temperature to above 200°C [6].

Alloy	Composition	Transformation range $(^{\circ}C)$
$Ag - Cd$	44–49% Cd	-190 to -50
$Au - Cd$	46.5–50% Cd	30 to 100
Cu–Al–Ni	14–41.5% Al; 3–4.5% Ni	-140 to 100
$Cu-Au-Zn$	23–28% Au; 45–47% Zn	-190 to 40
$Cu-Sn$	15 at % $\%$ Sn	-120 to 30
$Cu-Zn$	38.5-41.5% Zn	-180 to -10
$Cu-Zn-A1$	$3 - 8\%$ Al	0 to 150
	4-6% Al; 22-28% Zn	Room temperature
$In-Ti$	$18 - 23\%$ Ti	60 to 100
Ni–Al	$36 - 38\%$ Al	-180 to 100
$Ni-Ti$	$49 - 51\%$ Ni	-50 to 110
Fe-Pd	30% Pd	-100
$Fe-Pt$	25% Pt	-130
$Mn-Cu$	5-35% Cu	-250 to 180
Fe-Mn-Si	32% Mn; 6% Si	-200 to 150

Table 1.1: SMA activation temperature due to material composition (used without permission) [9].

In some cases, altering the physical shape of the SMA will also change the temperature at which it activates. This is further explored in section 3.4 on Frangibolts.

1.3 Project Statement

The ultimate goal of the project is to take adequate samples to analyze the microbial environment of holes 504B and 896A [2]. Due to the extreme environment of borehole 504B, a thermo-mechanically actuated sampler utilizing SMAs was designed, fabricated, and tested. Seven modular SMA triggers compatible with the samplers were designed, with four of these triggers fabricated for use in 504B and 896A for July 2019. All triggers that were sent on expedition were successfully tested at either SCU or MBARI. The design and testing processes are further outlined in the following sections. Preliminary designs for a dual-chamber gas-tight sampler was also developed for future models.

Chapter 2 System Design

Due to the extreme nature of hole 504B and the precise goals of expedition 385T, existing samplers in industry do not meet all of our standards. A set of parameters was established and compared to existing samplers to find ways of designing an adequate sampler that would be completely temperature dependent. This would also have to be accomplished with the SMA configurations available to the project as custom SMA configurations can take months to years to develop alone.

2.1 Existing Samplers

There are many different types of samplers used for fluid extraction in oceanic studies. Many of these samplers are used for seafloor applications while a fraction can be used for deep sea boreholes. While nearly all the samplers themselves fit in the borehole's size constraints, their actuation methods typically either exceed the borehole's size constraints or could be damaged when lowered into the borehole.

2.1.1 Niskin Sampler

For deep-sea water sampling, the most commonly used style of sampler is the Niskin sampler. Niskin samplers have two ends with caps that are left open while the sampler is lowered down to its sample, meaning that water flows through the sampler on the way down. Once the sampler reaches its target depth, the operator sends a signal down the wire to actuate a mechanism that closes and seals both caps. For these samplers, the operator decides to send the signal based off the temperature readings from a thermometer or thermocouple. The drawback of using this sampler is that as the sampler lowers, particles from the higher sections of the borehole could attach to the

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walls of the sampler, introducing uncertainty on what particles are present at specific depths.

Figure 2.1: Niskin samplers in a "rosette" formation (used without permission) [10].

2.1.2 Kuster Flow Through Sampler

Another type of common sampler used in deep sea research are Kuster Flow Through samplers (FTS). These samplers, like the Niskin sampler, also feature open valves on both ends, allowing fluids to flow through the sampler while traveling down the borehole. The two valves are spring-loaded and tripped by a timer on the surface, meaning the sampler must be tethered to the ship lowering it down. As this sampler only uses time as a means of actuating, researchers can only estimate the temperature of the water sample at time of collection by extrapolating data of the temperature gradient of the borehole [11]. For the purpose of this project, the temperature of the sample is more crucial than the time at which it is taken, so this time-activated style sampler would not work. It also has the same problem of the Niskin sampler of exposing the inner walls of the sampler to water as it travels down the borehole.

Figure 2.2: Kuster flow through sampler, with the "bottom" of the sampler to the left and "top" of the sampler on the right (used without permission) [11].

2.1.3 Walden Sampler

The sampler designed in this thesis is most similar to the Walden sampler developed in 1979 by Barrie Walden. This sampler is a syringe-type titanium sampler that utilized a spring and pin to take a water sample. A hydraulic arm was used to pull the pin, which then allowed for a second spring to the pull the piston. The sampler also has a snorkel to target hydrothermal vents more precisely [12]. The syringe aspect of this sampler makes it ideal for experiments that require a low probability of contamination from the surrounding environment.

2.2 Product Specifications

2.2.1 Borehole Specifications

Borehole 504B is a three-kilometer-deep borehole off the coast of Chile in the Pacific Ocean. The borehole begins at the ocean's seafloor with a 12 inch diameter opening. The drillship assembly is lowered into the opening, where the borehole tapers down to about four inches in diameter.[3] As such, samplers and all of their components must fit within the narrow channel. To account for ship movement, samplers are designed to be 3.5 inches in external diameter with no components sticking out past the outer wall of the sampler's body. All screws were countersunk to stay flush with the sampler. The narrow borehole also means the samplers must be strong enough to maintain their own weight in addition to the 1000 pound sinker bar attached to the very bottom of the assembly, as there is no space for structural support to be attached to the samplers. Couplers are also designed and fabricated as a means of attaching the samplers together. Temperatures in 504B begin at around 2°C at the top of the borehole and reach as hot as 200°C when nearing the bottom of the hole. For Expedition 385T, shape memory alloys with activation temperatures of 80°C, 90°C, 100°C, 140°C, and 170°C were

selected to design with.

Figure 2.3: Bottom hole assembly (BHA) of a typical drillship's assembly (used without permission) [13].

2.2.2 Biological Requirements

As the objective of the expedition had a biological research component, all materials that could come in contact with the sample must have antibacterial properties. All samplers must be easy to clean prior to deployment by cleaning staff on deck of the ship, meaning the design would have to allow for multiple uses and be easy to reset by a person with regular tools while also not reacting to cleaning solutions. The sample would also have to be easy to retrieve from the sampler, either via a valve or another opening. All materials would also have to be able to withstand the high borehole temperatures without degrading and thus contaminating the specimen. Additionally, to collect a large enough sample to test for microbes, samplers must be able to capture at least one liter of water.

For these reasons, all metal components that can come in contact with the sample were constructed of either titanium alloy Ti-6Al-4V or grade 2 titanium. O-rings and other

soft fittings were made of corrosion-resistant polymers, such as Viton and high grade silicone. All fasteners have hex heads to avoid the need for excess tools; one hex key set is all an operator would need to reset these devices.

2.3 System Requirements

Given the environmental and biological requirements of the expedition, the following requirements were determined for the sampler and mechanism system:

- 1. Sampler Body
	- a. Diameter must be less than 3.75 in.
	- b. Internal components, including mechanism, cannot extend past the outer wall of the sampler.
- 2. Integration
	- a. Samplers must interface with the IODP drillship attachments.
	- b. Samplers must support their own weight as well as the weight of the sinker bar attachment.
- 3. Temperature
	- a. Samplers and all internal components must be able to withstand up to 200°C.
	- b. Mechanism will be actuated purely by thermal energy of surrounding water.
	- c. Mechanism must demonstrate repeatability within $+/- 5^{\circ}C$
		- i. At least three successful trials are required to demonstrate this repeatability
- 4. Material
	- a. Samplers and all internal components must be able to withstand up to 10,000 psi.
- b. Samplers and all internal components must be made from corrosionresistant, anti-bacterial materials.
- 5. Sample Volume
	- a. Sample chamber must be at least 1 liter.
- 6. User Interface
	- a. Samplers must be easy to open, close, and reset by hand.

2.3.1 Subsystem Breakdown

The sampler is broken down into four major subsystems: external body, piston, nozzle, and mechanism. The external body subsystem consists of the outer cylinder, couplers, and drillship attachments. The piston subsystem consists of all internal components that comprise the syringe portion of the sampler, excluding intake. The nozzle subsystem contains all the components for sample flow. The mechanism subsystem consists of the trigger mechanism and fasteners to the sampler. Figure 2.4 shows the bounds of the four subsystems below:

Figure 2.4: Subsystem breakdown of full sampler assembly.

The subsystems are further described in detail in Chapter 4.

Chapter 3 Trigger Design

3.1 Overview

Several triggers were designed with different configurations to best utilize the SMA's available. Since the samplers are meant to be used for any temperature, the triggers all had to fit within the cutaway in the sampler body. All triggers also had to be fastened to the sampler body in the same way and latch onto the same notch in the piston rod.

3.1.1 Conventional Sampler Actuation

Typically, sample valves for existing closed-before-intake samplers are actuated via hydraulic rams located on a manipulator arm of the submersible the sampler is attached to, and as such is normally used in open environments [14, 17]. Open samplers, such as Niskin or Kuster samplers, are tripped using a cable that reaches either a timer or an operator at the drillship end [10, 11]. Self-triggering actuators can be achieved with the use of battery packs if there is enough space in the environment in which the device is being sent to, such as in seafloor applications.

Because of the narrow diameter of the deep-sea borehole, options to package an electronic actuator would be difficult without increasing the overall length of the sampler to accommodate the extra volume required. Additionally, electrical connections can be susceptible to damage by the high temperature of the water and potential exposure to the water itself. Servos and other motors that control valves would need to have protected cables so that damage will not occur while traveling down the borehole. Oil-filled bags or compartments are common insulators for electronics used in liquid environments, which is another potential failure point.

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3.1.2 Electronic Temperature Sensors

For most temperature-sensitive applications, electronic thermometers or thermistors are usually used to signal either an operator or a controller to actuate a device. For deep-sea boreholes, the electronic sensor and trigger cable would have to be tethered to the drillship, meaning at least three to four kilometers of tether would have to be maintained for proper function. An example of concern-for-wear can be seen with IODP's CORK system from 1996, which utilized a 164 meter long cable of 12 twisted pairs of thermistors. The cable was housed in Kevlar and had a sealed lower end to prevent any potential fluid contact with the data logging electronics. This thermistor cable was used in Hole 1024C and was repurposed in 1999 for Hole 1200C. The only reason IODP could reuse this cable was because Hole 1024C had a maximum temperature of 30°C, far lower than 504B, and there was no damage during recovery. A system like this would not have survived a second use after 504B and could potentially stop working while deployed [15]. Additionally, active components run the risk of power failure during deployment. By using shape memory alloys, there is no need to even run an electrical current through it, like a passive thermistor or thermocouple. SMAs also eliminate the need for batteries and can significantly save space in the already cramped conditions.

3.1.3 SM A Availabilities

For this cycle of trigger development, four configurations of SMAs were considered: wire, tension springs, compression springs, and Frangibolts. Three general trigger styles were produced with these options in mind as the different configurations provide different advantages in terms of SMA performance.

Wires have low linear travel and can be susceptible to cracking under too much load. The amount of force produced through linear travel however is very high, making short but strong applications best for a wire configuration.

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Figure 3.1: [top to bottom] 5mm diameter CuAlNi blend, 2mm diameter CuAlNi blend, thin-wire NiTi.

Springs are great for longer travel but do not produce as much force as wires, meaning that this configuration is best for low-load applications. Because of the longer travel length, springs are easier to use than wires for most trigger applications within the scope of this project. SMA tension springs can be stretched while below the activation temperature and will retract upon heating. SMA compression springs can be compressed while below the activation temperature and will expand upon heating. The idea of creating custom springs using the 2mm diameter CuAlNi blend was explored briefly before determining risk of oxidation of the material would be too high.

Figure 3.2: [left to right] NiTi tension spring, high temperature compression spring, NiTiCu compression spring.

Frangibolts are a proprietary SMA configuration manufactured by TiNi Aerospace. These are essentially hollowed out cylinders made of SMA material that expand linearly upon heating to break a pre-notched fastener running through the cylinder. Oftentimes electronically actuated in aerospace applications to break connections, Frangibolt mechanisms in this project utilize the hot water as the means of heating and actuating the Frangibolt.

Figure 3.3: Frangibolt loaded in TiNi's proprietary reset tool.

3.1.4 Previous M echanism Design

In 2015, SCU student Thomas Hoye developed a thermally actuated mechanism for Niskin-style water samplers for use in deep-sea boreholes. This mechanism was designed for temperatures up to 90°C with potential for 270°C designs with further research. At the conclusion of the project, a working prototype of the design was constructed and tested [16].

Figure 3.4: Final design of Hoyes' thermally actuated mechanism (used without permission) [16].

The mechanism uses two torsion springs and two Dynalloy SMA tension springs with an activation temperature range of 70°C to 80°C. Testing of this mechanism demonstrated reliable precision of actuation around 60°C. However, this mechanism sits on the exterior of the sampler, which violates the design constraint of fitting all components and triggers inside the sampler itself to avoid potential damage from the borehole walls. Additionally, this design only works with tension springs, which limits the range of actuation temperatures.

3.2 Slider Trigger

The slider trigger was designed for the tension and compression springs available. Because the springs do not produce much force, all sliders needed to be as low friction as possible. Similar to Hoyes' mechanism, this design uses linear travel of an SMA to directly pull a tab out of locking the intake mechanism, in this case the plunger.

3.2.1 90°C Tension Spring

The 90°C tension spring is a NiTi alloy produced by Dynalloy. The spring is produced from wire of 0.020 in. diameter and coiled at a diameter of 0.136 in. The pull force of spring when heated is about 0.5 lbs. Four springs are attached in parallel to each other from the baseplate of the mechanism to the edge of the slider. Two stainless steel

tension springs keep the slider in the "closed" position, where the slider sits in the notch in the piston rod. When the SMA springs are heated, they overcome the tension force of the stainless steel tension springs and friction force of the slider in the notch to become "open." The plunger is then able to move upwards and take a sample.

Figure 3.5: [left] tension spring design in "closed" position, [right] tension spring design in "open" position.

While testing prototypes, it was determined that the friction of the steel slider on the titanium rod of the plunger was too high for the SMA springs to overcome. One prototype was thus coated in Teflon while another was coated in Tungsten Disulfide (WS2). The WS2-coated mechanism operated with better performance but poor repeatability as the coating corroded away faster in the hot water. The Teflon-coated mechanism had less ideal results but lasted longer. It was concluded that these coatings would be too unreliable in a high-stakes deployment, and this design was ultimately left out of final production.

3.2.2 80°C and 100°C Compression Springs

Both the 80 and 100 compression springs were manufactured by SAES Group. The 80°C spring is made of a NiTiCu blend with a natural length of 0.64 inches and can be compressed down to 0.36 inches. Because the force output of the 80C compression spring is relatively low at roughly 1.8 lb, two SMA springs had to be used for the slider design, bringing the overall force output to about 3.6 lbs. A stainless steel tension spring runs co-axially to the SMA springs to keep the mechanism in the "closed" position before deployment.

Figure 3.6: [left] 80°C compression spring design in "closed" position, [right] 80°C compression spring design in "open" position.

The 100°C compression springs are similar to the 80°C compression springs in travel and force output. These springs are made of a proprietary blend of alloy and have a wire diameter of 1.50 mm and coil diameter of 10.3 mm. This design also utilized two compression springs, with a total force output of about 6.7 lbs. Like the 80°C configuration, this design also had stainless steel tension springs run co-axially to the compression springs.

Figure 3.7: [left] 100°C compression spring design in "closed" position, [right] 100°C compression spring design in "open" position.

3.3 Ball Bearing Trigger

The ball bearing design was developed to translate the small force of one compression spring into a larger movement of two flippers letting go of the piston rod. The compression spring sits under a small plate that contains two pins. These two pins sit inside holes in the two flippers to keep them "closed." When the SMA is activated, it pushes the plate up which thus pulls the two pins out of the two flippers. The notch on the rod, with a slight bevel, pushes against the ball bearings press-fitted into the

flippers, pushing the flippers out of the way, and the rod is then free to slide up the sampler to take a sample.

Figure 3.8: [Left] ball bearing mechanism in "closed" position, [right] ball bearing mechanism in "open" position.

This mechanism had iterations with both compression springs and the 90°C tension springs. Prototypes of the 90°C tension springs failed to provide results adequate enough for reliable usage, so only the compression springs moved on past the testing phase.

Figure 3.9: Ball bearing mechanism with 90°C adapter and two 90°C SMA springs.

3.4 Frangibolt Trigger

The Frangibolt trigger utilizes one Frangibolt created by TiNi Aerospace at a time. The trigger design is compatible with both the 140°C and 170°C Frangibolts. Both

Frangibolts use the same material composition; the only variance between the two are dimensions. This trigger is slightly less modular than the other designs, as a custom bracket has to be fitted on the piston rod while assembling the samplers. The sampler is set by lowering the plunger (with the bracket attached to the rod) down the sampler and bolting the bracket to the Frangibolt half of the trigger. The Frangibolt half is bolted to the guideplate as seen in Figure 3.8.

Figure 3.10: Frangibolt trigger with all sampler components hidden.

The Frangibolt trigger works by expanding when it reaches its activation temperature, snapping the fastener holding the bracket down, and thus releasing the rod to take a sample. The Frangibolt is recovered while the fastener is discarded once the trial is done.

For the testing phase of this project, MBARI manufactured Frangibolt-style SMAs from 5mm CuAlNi stock material. This is further explored in Section 5.2.
Chapter 4 System Design

The sampler and remaining components had to be designed to be self-contained and structurally stable when connected to the drillship assembly. Drawing inspiration from other samplers mentioned in Chapter 2 while also using a previous design by MBARI, a final sampler was designed and fabricated. Testing results can be found in Chapter 5. The following sections detail the different sections of the sampler assembly, divided into Sampler Body, Plunger Sub-Assembly, Valve Sub-Assembly, Mechanism Interface, and Constant Force Spring Assembly.

4.1 Sampler Body

4.1.1 Initial Designs

A previous syringe design by MBARI was created for a different expedition in the past. This sampler has a much smaller sample chamber than our necessary sample volume, so this syringe would not work as designed, but many qualities were taken and expanded on for the initial and final iterations.

Figure 4.1: Previous syringe sampler designed by MBARI.

Initial designs of the sampler started with the previous design as inspiration. The sampler was designed so that instead of two brackets attaching to the tubular chamber wall, our sampler wall would be made from titanium and be threaded to connect to other samplers or drillship assembly components. Since our sampler would be going to pressures reaching 13,000 psi, the sampler would need cutaways in various spots to allow for water to flow into empty spaces, eliminating the risk of air pockets. Because of this, the floating piston seen in the previous design would not work. Our piston would feature a piston rod and be pulled by some sort of tension, later determined to be a constant force spring. The valve assembly seen at the bottom would then be modified to fit inside the titanium sample and not fix onto a separate bracket as seen in Figure 4.1. The edge of the sampler would have to extend well past the nozzle so that another connector could screw onto it.

4.1.2 Final Design

The sampler body was designed to be compatible with all of the mechanisms that were developed. This meant designing a Mechanism Access slot large enough for all designs to fit as well as notching the rod with the correct size and slight bevel. The body of the sampler was made entirely of titanium to both be antibacterial and structural support for the drillship assembly. Intake slots were designed to allow for maximum flow to the intake nozzle. However, since the samplers travel relatively slow down the borehole, the intake slots do not need to be as large. Small slots were cut into the upper portion (seen to the left in Figure 4.1) to allow for water to equalize pressure in the sampler. All sampler body holes that were meant for screws were countersunk to minimize protruding screws while going down the borehole. All major inner structures, which are the constant force spring's holder, the middle guide plate, and intake valve, use the same ¼"-20 size screws for uniformity. Two long, thin slots were cut in the sides of the sampler to serve as guides for the T-cross of the piston system in order to prevent angling of the piston as it travels to take a sample. The ends of the samplers were

threaded with for 3-5/32"-8 ACME thread to be compatible with any of the three connectors.

Figure 4.2: Full sampler assembly with marked sub-sections; left connector is the "top" connector to the drillship while the right connector is a standard coupler to attach another sampler.

Three connectors were developed for interfacing the sampler body with the drillship assembly and other samplers. Sampler-end threads of the connectors were all standardized to 3-5/32-8 stub ACME threads, and the inner, wider cross section was designed for drillship equipment used for torqueing down the sinker assembly.

Figure 4.3: [from left to right] Sampler-to-sampler coupler, sampler-to-ship connector, sampler-to-sinker bar connector.

4.2 Plunger Sub-Assembly

4.2.1 Initial Design

The initial design of the plunger featured a double o-ring plunger head with a square groove near the top of the plunger for the mechanisms to slot into. The double o-ring configuration was designed first as there are other syringe-style samplers designed in the past using two o-rings, such as in gas-tight applications which will be further explored

later. After consulting with Hans Jannasch of MBARI, the double o-ring design was changed to a single O-ring to avoid the possibility of a vacuum forming between the two o-rings and therefore causing too high of static friction for the constant force spring to overcome. The plunger rod would also have to incorporate a way of fixing onto a spring to be pulled for sample intake. Original designs focused on finding a tension spring to fit the needs of pulling the plunger up the sampler, which is further described in section 4.5.

4.2.2 Final Design

A plunger was designed to pull in water during extraction. The plunger head uses a high temperature silicone o-ring to withstand repeated use in the borehole's corrosive environment. The notch towards the top of the plunger has a slight bevel on the lower edge, just enough for the ball bearing mechanism to push against but not enough that the sliding mechanisms could not work. A thin rod runs perpendicular to the plunger rod to travel along the thin guide slots of the sampler. Since the plunger head contacts the sample, the final plunger heads were constructed of titanium. The rods were also made of titanium while the cross rod was made of stainless steel. The cross rod also attached to a yoke for the constant force spring to attach to plunger, allowing for pivots as the plunger travels through the sampler. A rulon bushing press-fit into the middle guideplate of the sampler provides a low-friction interface for the plunger to stay centered to.

The sample chamber measures 8.954 inches in length with a 3.068-inch diameter. This equates to a sample volume of 66.19 cubic inches, or 1.08 liters.

Figure 4.4: Final plunger rod and head with silicone o-ring and cross rod.

4.3 Valve Sub-Assembly

4.3.1 Previous Valve System

A previous nozzle designed by MBARI was used as a base for the final nozzle. This nozzle was made of titanium for another microbial experiment and featured a square bracket and clear polycarbonate chamber sandwiched between its square bracket and another square bracket at the top of the sampler. The nozzle's plug hole was threaded for a 5/32" hex drive plug. A valve stem sits inside with a compression spring to make the nozzle flow in only one direction. This concept is kept for the final design of our project's sampler as we do not want any sample escaping as the assembly is pulled up out of the hole.

Figure 4.5: Previously designed nozzle on syringe.

4.3.2 Final Valve System

The custom one-way nozzle consists of all-titanium parts with a Viton flat washer to seal the intake. No components of the nozzle could be constructed from stainless steel due to microbial constraints. Another high temperature silicone o-ring sits in a notch running around the exterior of the circular valve to seal the sample into the sample chamber. The originally $5/32$ " plug was changed to be a $\frac{1}{4}$ "-20 titanium plug as $\frac{1}{4}$ "-20 is a more standard and readily available size to find. The plug sits on the exterior of the nozzle for sample extraction, and a titanium spring sits around the valve stem. Due to the modification in plug size, rubber washers and their slots had to be slightly modified to avoid interferences. The valve assembly was also modified to have a round exterior to replace the square bracket of the previous model.

Figure 4.6: [from left to right] top-front view of assembly, bottom-side view of assembly with plug visible, crosssection of assembly.

4.4 M echanism Interface

The mechanism access slot measures 2.77 in. by 1.26 in. This allows for triggers to have plenty of space to be inserted and reset by hand. The slight bevel on the bottom of the rod's notch is visible in Figure 4.4. All mechanisms except for the Frangibolt mechanism are screwed into the sampler body by two 6-32 screws on the opposite side of the mechanism access slot, seen in Figure 4.4 in the rear of the figure. A 10-32 screw in the mechanisms themselves sit in the mechanism access slot to keep that side of the mechanism fixed to the sampler.

Figure 4.7: Close-up of mechanism access slot with plunger rod and yoke for reference.

The Frangibolt design uses the inner guide plate as its base to fix onto. Two screws travel through the Frangibolt-holding part of the mechanism while the plunger adapter connects to the Frangibolt. This way the Frangibolt-holding part stays attached to the inner plate even after the Frangibolt breaks the connection to the plunger rod. To accommodate the Frangibolt, the inner plate had two holes threaded for the two screws.

Figure 4.8: Frangibolt mechanism mounted to inner guide plate.

4.5 Constant Force Spring Assembly

As mentioned previously in section 4.2, a standard corrosion-resistant stainless steel tension spring was considered to pull the plunger to take a sample. However, two primary concerns of pursuing this were finding a spring with a long enough travel to take the full sample while also keeping the fully extended spring's force low enough that the SMA mechanisms could overcome friction, which could also be problematic as the spring retracts the force could become too weak to take in a sample. Due to these concerns and troubles finding adequate springs, a constant force spring was selected to be designed with.

Once it was decided to use a constant force spring, three dimensionally similar springs with different force values were selected to design for: 24 lb, 28 lb, and 32 lb. A stainless steel spool with cross-rod was designed to screw into the walls of the sampler for the constant force spring to sit on. The constant force spring stays on the spool due to the force of the spring itself tightening onto the spool. The end of the spool is connected onto a custom designed yoke that attaches to the plunger rod by running its crossrod

through the yoke. This yoke is able to swivel about the rod to allow the constant force spring to pivot as it travels up the sampler. The 28 lb constant force spring was selected as the final spring after numerous dry and wet runs with the 24 lb and 28 lb springs. The 32 lb spring was too difficult to uncoil and fit onto the spool, and the 28 lb spring was strong enough anyways.

Figure 4.9: Constant force spring sub-assembly with retracted spring.

Chapter 5

Testing

Testing of the mechanisms focused on repeatability and precision. The target precision of actuation was $+/- 5^{\circ}$ C while repeatability centered on the components of the sampler working as intended for multiple uses. These standards apply to both low temperature and high temperature mechanisms, so all testing will be determined as successful if they meet the same performance criteria.

5.1 Low Temperature

"Low Temperature" refers to any of the SMA configurations that could be tested using water as a fluid. The designs that made it to the testing phase were the 80°C and 100°C compression springs and 90°C tension spring in the ball bearing mechanism.

All trials were conducted in a 6 in. by 8 in. by 36 in. stainless steel tank set on top of two electric burners. The samplers were propped up in the tank by wood two-by-fours to prevent conduction from the hotplates. Room temperature water was then poured into the tank to cover the entire sampler, and three thermocouples were placed at three different positions on the sampler. The three positions were midway down the tank for ambient tank temperature, inside the mechanism chamber for the SMA's temperature, and right outside the nozzle for intake temperature. This was to show the temperature distribution of the tank to better demonstrate the SMA's triggering behavior.

Figure 5.1: Sampler with three thermocouples attached in water bath with covers to quicken heating process.

Once setup was completed and the thermocouples were attached, the hotplates were turned onto "high" and temperature was tracked until movement occurred. At the end of every trial, the water was cooled to about 60°C and scooped out to retrieve the sampler. The sampler was then reset by taking out the mechanism, squishing the compression SMA's or extending the tension SMA's back into place, and reinserting the mechanism into the sampler. The sampler's plunger is also reset back into its undeployed position while reinserting the mechanism. The sampler is set back into the tank, room temperature water is poured in, and the process repeats for all trials. All results were tabulated and can be found in Appendix C. A trial was deemed "successful" if the mechanism properly pulled the pins from the flippers to allow the rod to move and the sampler took a full water sample. Successful trials also had to have actuation within 5°C of the resultant average of their SMA's subset of trials. The SMA manufacturer's activation temperature is used as an estimate for actuation, but the accuracy of the mechanism to that manufacturer's value is not as important as the repeatability of the mechanism itself. A "partial" success meant that the mechanism worked properly but for other reasons the sampler was unable to take a full sample, such as the constant force spring scraping the insides of the sampler or hard water buildup on the constant force spring holder preventing it from spinning. A "no" success means that the mechanism itself failed to actuate.

Ball Bearing: 100°C SMA Compression Spring									
Test#	Mechanism Temp [°C]	Deviation from Average [°C]	Performance						
5	90.63 -2.69		Successful						
6	96.86	3.54	Successful						
$\overline{7}$	93.82	0.5	Partial						
8	91.01	-2.31	Successful						
9	94.24	0.92	Partial						
10	93.41	0.09	Successful						
Average	93.32 1.68								
Standard Deviation	2.29	1.37							
Ball Bearing: 80°C SMA Compression Spring									
Test#	Deviation from Average [°C] Mechanism Temp [°C]		Performance						
11	78.36	-2.45							
12	79.48	-1.33	Successful						
13	81.52	0.71	Successful						
14	78.09	-2.72	Partial						
15	80.63	-0.18	Partial						
17	85.3	4.49	Partial						
26	81.22	0.41	Partial						
27	82.43	1.62	Partial						
28	80.34 -0.47		Partial						
Average	80.81	1.59							
Standard Deviation	2.2	1.41							
Ball Bearing: 90°C SMA Tension Spring									
Test#	Mechanism Temp [°C]	Deviation from Average [°C]	Performance						
16	91.4	-1.92							
19	94.24	0.92	Partial						
20	94.67	1.35	Partial						
24	93	-0.32	Partial						
Average	93.32 1.12								
Standard Deviation	1.47	0.68							

Table 5.1: Compiled results of Low Temperature trials.

As seen in Table 5.1, all trials met the criteria of remaining within $+/- 5$ degrees of each other. The 90°C tension spring design had the same average actuation temperature as the ball bearing design, so these trials were ended early in favor of conducting more trials of the 100°C ball bearing design for more evidence of repeatability. Trials 1, 2, and 4 were omitted from the tables as they were with a different mechanism, and 18, 21-23, and 25 were omitted as they had improper or no mechanism actuation and therefore did

not have a mechanism temperature to report. Trials 18, 21-23, and 25 are included in Figure 5.2.

Figure 5.2: Mechanism and sampler failures of the same sampler across 28 trials.

Over the course of the two months testing took place, it appeared that the constant force spring was having problems first with friction in the spoolholder and then eventually with the sides of the spring scraping against the inside of the tube. While mechanism performance was rather stable for the 80°C and 100°C trials, the sampler's performance progressively got worse as testing continued, evidenced in portions of Figure 5.2 where the mechanism curve is flat while the sampler curve has a slope of 1. While it's presumed that the hard tap water reacting with the aluminum components may have caused considerable buildup, there is still potential problems with the orientation of the constant force spring as it does come into contact with a considerable portion of the sampler's inner wall, especially when fully extended. Given that the sampler made it through most of its trials without getting considerably stuck, the samplers could probably last for a few expeditions before needing to retire. That being

said, for longer term usage in the range of decades, a different method of mounting the constant force spring or the addition of a shield could perhaps be useful.

5.2 High Temperature

"High temperature" refers to mechanisms that need a fluid other than water to be tested while at atmospheric pressure. All high temperature tests were conducted at MBARI. A slightly smaller, aluminum tank was filled with cooking oil. The reset sampler was placed in the oil bath and then slowly heated up to temperature. The triggers tested were MBARI-made "Frangibolt"-style triggers made from 5mm CuAlNi rod by boring the inside to fit a notched fastener. Each specimen was cut at 0.6 inches in length and were tested for repeatability. As the temperature is not as important as repeatability, this testing data serves to categorize the activation temperature of the SMA triggers rather than compare the experimental values to theoretical values. Only one temperature reading was taken for the high temperature tests analogous to the "ambient temperature" readings of the low temperature tests.

	Specimen Activation Temperature [°C]								
Trial	\mathbf{A}	B	$\mathbf C$	D	E	F	G	H	
1	171.1	172.1	159.6	132.7	130.3	127.0	105.2	107.3	
$\overline{2}$	176.1	184.8	166.2	135.6	123.0	122.8	102.3	106.4	
3	175.8	184.8	158.6	139.1	125.2	133.5	100.4	107.7	
$\overline{4}$				136.5	127.1	129.8	101.2		
Average	174.3	180.6	161.5	136.0	126.4	128.3	102.3	107.1	
Std Dev	2.8	7.3	4.1	2.6	3.1	4.5	2.1	0.7	
Trial	Specimen Deviation from Average [°C]								
	A	B	$\mathbf C$	D	E	F	G	H	
1	3.2	8.5	1.9	3.3	3.9	1.3	2.9	0.2	
$\overline{2}$	1.8	4.2	4.7	0.4	3.4	5.5	0.0	0.7	
3	1.5	4.2	2.9	3.1	1.2	5.2	1.9	0.6	
$\overline{4}$				0.5	0.7	1.5	1.1		
Average	2.2	5.6	3.2	1.8	2.3	3.4	1.5	0.5	
Std Dev	0.9	2.4	1.5	1.6	1.6	2.3	1.2	0.3	

Table 5.2: Compiled results of High Temperature trials.

As seen in table 5.2, specimens B and F had trials that did not meet the $+/-5^{\circ}$ C limit from the average (highlighted in yellow). Specimen B had an average deviation of 5.6, above the 5°C threshold and therefore too unreliable for an accurate temperature reading. However, even though the temperature range for this specimen were larger than desired, the mechanisms and sampler were still functional over repeated use. As these SMA's were not machined in a professional machine shop nor by a machinist with SMA expertise, there is a chance of deformations in the material causing the wider range of actuation temperatures. TiNi Aerospace's Frangibolts are aerospace grade with tolerances of $+/- 0.002$ inches. The "Frangibolts" tested here were to the nearest 0.01 inch with unspecified finishes.

It was also reported during these tests that similar problems of the piston getting stuck occurred at MBARI as well even with the oil medium. While MBARI was able to fix the problem by flipping the orientation of the constant force spring, it is unclear if the sampler is suitable for high-cycle use.

Chapter 6 Conclusion

6.1 Summary

The objective of producing a reliable SMA-actuated sampler compatible for use in deepsea boreholes for designated temperatures with no electronics was met, along with several SMA triggers. While out at sea on Expedition 385T, both boreholes 504B and 896A were plugged with packers from previous expeditions, preventing any sampler deployments. However, these samplers can still be used for future expeditions to boreholes of similar temperature ranges. The mechanisms can be modified to work with other SMA's, and the large mechanism access slot can accommodate more types of mechanisms if necessary.

The success from this project will help direct further iterations of the sampler and triggers, with possibly more new blends and configurations to design with. It has also laid a foundation for more joint work between university research assistants, IODP, and MBARI for advancements in borehole research. Shape memory alloys provide a new avenue for developing exploratory tools, leading the way to new and exciting discoveries in deep-sea boreholes. Specifically, marine scientists can use these samplers to better catalog sub-seafloor life in deep-sea boreholes and further test the thermal limits of life on the planet. All low temperature and most high temperatures demonstrated strong repeatability and precision, with temperature deviations remaining within a 5°C threshold. By utilizing samplers that react to specified temperatures, scientists can now accurately determine what life forms can exist at what temperatures and could potentially find life in places previously unexplored.

6.2 Future Work

Future work stemming from this project are gas-tight sampler development, exploration of new SMA blends, and smaller-scale electronically actuated samplers.

6.2.1 Gas-Tight Applications

A gas-tight sampler utilizing an inert-gas chamber for pressure stabilization while extracting samples with dissolved gases. Based off a sampler developed by Jeff Seewald, this dual-chamber sampler will be actuated by a shape memory alloy rather than the usual electric signal.

Figure 6.1: Cartoon schematic of J. Seewald's sampler depicting pre-deployed, post-deployed, and retrieval stages [17].

The sampler uses inert nitrogen gas to keep the contents of the sampler at constant pressure. To prepare the sampler, the gas chamber is pre-charged to about 10% of the expected final pressure with an inert gas; in this case, nitrogen was used. The rest of the sampler is filled with seawater as a buffer between the gas and sample water. Upon reaching the target location, a signal from the ROV triggers the servo motor to open the intake valve. The sample is taken for a set amount of time and then the servo motor closes the valve, ensuring no contamination of the sample. To retrieve the sample, a

pump is used to relieve the pressure in the gas chamber and then the sample is extracted through a micrometering valve attached to the other chamber [17].

The Seewald sampler does not meet this project's design constraints as there are several components on the exterior of the chamber. These components would either not fit in the borehole or would be severely damaged if they came into contact with the borehole walls. Because of this, finding a valve that fits in the narrow borehole as well as fabricating an elaborate enough open-and-close one shot mechanism would be the main challenges of this new venture.

Preliminary designs for proof-of-concept were developed during the second year of this project for further development in later years. The design follows that of Seewald with the dual chamber design and gas pre-charge section. The pre-charge system has yet to be determined, but a hole has been placed in the chamber for interfacing. The two chambers twist onto the inner coupler which contains a narrow channel for fluid flow. This channel will eventually be replaced by a Viscojet flow regulator to ensure a slow intake and therefore prevent any microbial damage or dissolution of gases. Double Oring pistons are free-floating in the two chambers, with their movement determined by the flow restrictor. For intake, few needle valves exist that fit in the borehole. Autoclave Engineers' Low Pressure, Single Ferrule 10V2 and SW Series angle needle valve appears to be the best fit for this application, but there are also potential solutions in other types of valves, such as bellows valves or ball valves. Further documentation on dimensions and parts can be found in Appendix B.

Figure 6.2: CAD mockup of proof-of-concept design in pre-deployed [top] and post-deployed [bottom] configurations.

Figure 6.3: Schematic of Autoclave Engineers' angle needle valve [20].

For opening and closing the sampler in one smooth motion while timed with full extraction, some gear train possibilities were explored. One promising concept was a reversible gear train achieved by using a planetary gear train with some teeth removed on the sun and ring gear.

Figure 6.4: Reversible gear train mockup.

With further development, an SMA could trigger a torsional spring to drive the reversible gear train and therefore open and close the sampler's valve. Factors such as timing and pressure would have to be considered.

6.2.2 New SM A Blends

This project has potential access to new SMA blends that have not existed before through partnerships with metallurgists and TiNi Aerospace. As the new blends are made available, further materials testing and design work can be conducted. In 2019, a senior design team at SCU created a thermal testing chamber to test the 5mm wire mentioned earlier, as when the material was received it had yet to be properly characterized [21]. By conducting further tests on new blends to test if they can produce enough force or withstand enough heat, more potential temperature ranges can be explored.

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Appendix A: Mechanism Drawings

A1. Slider Mechanism – 90°C

A2. Slider Mechanism – 80°C

A3. Slider Mechanism – 100°C

A4. Ball Bearing Mechanism

A5. Frangibolt Mechanism

Appendix B: Sampler Drawings

B10

B13

Bill of Materials – Ordered Parts

Appendix C: Testing Data

C1. 90°C Ball Bearing Mechanism

C2. 100°C Ball Bearing Mechanism

Test#	BB80 Test #	Date		Successful? Actuation Temp [°C]	Notes
					The mechanism worked without the sleeve around the SMA spring. However, the
11	$\mathbf{1}$	7/10/2019	Partial	78.36	piston did not rise at all and was very stiff even with assistance. Piston movement
					seems to be getting worse.
12	$\overline{2}$	7/12/2019	Yes	79.48	This test was run with vinegar in the tank to try to solve the prblem of the piston
					sticking. This in combination with the fixing/flipping the yoke connect seems to have
					solved the problem. The SMA did not have a sleeve around it.
13	3	7/15/2019	Yes	81.52	This test was successful with both triggering the mechanism and full piston movement.
14	4	7/15/2019	Partial	78.09	This test had successful triggering of the mechanism but the piston got stuck about one
					inch away from full travel.
15	5	7/15/2019	Partial	80.63	This test had successful triggering of the mechanism but again the piston got stuck
					about one inch away from full travel.
17	6	7/16/2019	Partial	85.3	The full valve assembly was used with a stand-in spring, however it was found that this
					spring constant was way too high and the piston could not rise. The mechanism was
					successfully triggered
26	7	7/26/2019	Partial	81.22	The mechanism actuated as expected, and the piston moved quickly at first. However,
					the piston got stuck a couple inches from the top again.
27	8	7/26/2019	Partial	82.43	The mechanism was triggered, but the piston only went about two-thirds of the way up
					the slot. The constant force spring felt stiffer when pushing down to reset the
					mechanism.
28	9	7/26/2019	Partial	80.34	The mechanism was triggered, and the piston only traveled a couple inches. After
					removing the sampler from the water, the piston was forced up and scraping could be
					heard from inside the sampler.

C3. 80°C Ball Bearing Mechanism

C4. Frangibolt-Style Mechanism

Trial	Specimen Activation Temperature										
	A	B	C	D	Е	F	G	$\bf H$			
	171.1	172.1	159.6	132.7	130.3	127.0	105.2	107.3			
2	176.1	184.8	166.2	135.6	123.0	122.8	102.3	106.4			
3	175.8	184.8	158.6	139.1	125.2	133.5	100.4	107.7			
4				136.5	127.1	129.8	101.2				
Average	174.3	180.6	161.5	136.0	126.4	128.3	102.3	107.1			
Std Dev	2.8	7.3	4.1	2.6	3.1	4.5	2.1	0.7			