A New High Temperature Borehole Fluid Sampler (MTFS)

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Abstract

Deep (>1 km in depth) scientific boreholes are unique assets that can be used to address a variety of microbiological, hydrologic and biogeochemical hypotheses. Few of these deep boreholes exist in oceanic crust. One of then, Deep Sea Drilling Program Hole 505B, reaches ~190°C at its base. We designed, fabricated, and tested in the laboratory the Multi-Temperature Fluid Sampler (MTFS), a titanium syringe-style fluid sampler for borehole applications that is tolerant of such high temperatures. Each of twelve MTSF units collects a single 1-L sample at a predetermined temperature, which is defined by the trigger design and a shape memory alloy (SMA). SMAs have the innate ability to be deformed and only return to their initial shapes when their activation temperatures are reached, thus triggering a sampler at a predetermined temperature. Three SMA-based trigger mechanisms, which do not rely on electronics, were tested. Triggers were released at temperatures spanning 80°C to 181°C. The MTFS was set for deployment on International Ocean Discovery Program Expedition 385T, but hole conditions precluded its use. The sampler is ready for use in deep oceanic or continental scientific boreholes with minimal training for operational success.

1. Introduction

The current and future direction of scientific ocean drilling depends on technological advances to achieve a wide range of scientific objectives. Objectives related to microbial life in the subseafloor and a dynamic Earth represent two of the four current themes that guide scientific ocean drilling within the International Ocean Discovery Program (IODP) (IODP Science Plan for 2013-2023). While advances in these themes have been achieved from traditional coring and sample analyses, non-traditional means of instrumenting boreholes and direct sampling of legacy boreholes continue to transform our knowledge of these themes

(D'Hondt et al., 2019; Orcutt et al., 2011; Smith et al, 2011; Neria et al., 2016; Wheat et al., 2020). To meet new challenges afforded by future and legacy boreholes, including the potential for *in situ* manipulative experiments, a new arsenal of samplers and sensors need to be developed.

Scientific ocean drilling during the past five decades has resulted in >100 cased boreholes, many of which are suitable for re-entry and further discovery (Edwards et al., 2012). Such boreholes tap a range of thermal, hydrologic, physical, and crustal conditions, providing the underpinnings for a range of potential experiments to elucidate crustal and microbial evolution and function and the impact of both on ocean processes. Of special interest are the few deep boreholes that penetrate more than a kilometer below the seafloor, each taking many months to years to establish. As a result of natural geothermal heating from below, temperatures within such boreholes exceed 100°C with Deep Sea Drilling Project (DSDP) Hole 504B reaching temperatures >190°C at the base of the borehole, ~2000 meters below the seafloor (Guerin et al., 1996). Additional warm, deep boreholes exist in continental settings (e.g., KTB; Emmermann and Lauterjung, 1997) and in active high temperature hydrothermal systems (e.g., Brothers Arc Flux; de Ronde et al., 2019). To study *in situ* conditions within these challenging environments a new array of sensors and samplers need to be developed. Standard electronics do not tolerate such temperatures and, in general, do not function above 150°C without costly vacuum jackets (dewars) or cooling mechanisms. Because of (a) the uniqueness of these warm, deep boreholes, (b) the aspiration to characterize the thermal limits of life within the crust, (c) the desire to elucidate water-rock reactions and crustal alteration in a natural setting, and (d) the lack of a fluid sampler that is inexpensive, easy to operate, and affords a versatile array of experimental possibilities, we developed the Multi-Temperature Fluid Sampler (MTFS). The MTFS is a syringe-style fluid sampler that employs no electronics. Instead, it incorporates a mechanical trigger that utilizes the thermal-response properties of a shape memory alloy (SMA), which is a precise mixture of metals that affords the alloy to be physically modified at room temperature and returns to its original shape at an activation temperature that depends on the composition of the alloy, the geometry of the SMA material, and the design of the trigger mechanism.

2. Existing Samplers

Prior to the MTFS, borehole fluid samplers in the IODP inventory included the Water-Sampling Temperature Probe (WSTP), ~~and~~ the Kuster Sampler, and Schlumberger's single-phase fluid sample collection system. The WSTP has been used for decades (Mottl and Gieskes, 1990) and is lowered on a wire to the desired depth with a pre-set timer that opens the intake valve. The pressure differential between *in situ* pressure and 101 kPa~~ATM~~ drives fluids into the sampler, possibly lysing microbial cells. Only ~40 ml is collected in the sample tubing with ~1L spilling into a chamber that cannot be aseptically cleaned for trace metal and microbial determinations. The Kuster fluid sampler was most recently used on IODP Exp. 376 (Brothers Arc Flux; de Ronde et al., 2019). This sampler collects ~500 ml of borehole fluid during a single lowering and is closed by a mechanical clock. However, the sample container is open during deployment, potentially exposing the sample container to contamination (e.g., accumulation of grease, microbial mats, and other particulates). Neither sampler can be preloaded with acid, microbial preservatives, or metabolic tracers and only one sample can be collected during a single lowering.

~~Other m~~More complex samplers require dedicated technicians such as Schlumberger's single-phase fluid sample collection system that was used in conjunction with the Quicksilver In Situ Fluid Analyzer on IODP Exp. 337 (Inagaki et al., 2013). Multiple sample modules can be

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used, six samples were collected on IODP Exp. 337 (Inagaki et al., 2013). Other complex samplers include ~~are available through industry. For example,~~ Thermochem Inc.'s high temperature 2-phase downhole sampler is a vacuum jacket-type, memory tool and Leutert's positive displacement sampler and one phase sampler. The latter is a gas-tight system with an internal clock that opens and closes a valve. This system can collect a 0.6 L sample at temperatures to 180°C (Kampman et al., 2013). ~~Schlumberger's single-phase fluid sample collection system returns up to five modules of formation fluid in gas-tight samplers. The use of these samplers requires dedicated technicians.~~

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3. Design Criteria

The MTFS design was primarily based on the Walden-Weiss Titanium sampler (Von Damm et al., 1985) and the four borehole fluid samplers mentioned above. To meet the physical requirements of borehole use and ensure sample integrity, design criteria included the capability of the sampler to be (a) cleaned for trace element analysis, (b) aseptic prior to deployment, (c) tolerant of temperatures greater than 250°C, and (d) chemically inert but not gas-tight. Additional criteria for flexibility in sample recovery and experimental design included the capability of being primed with a reagent, such as acid to keep metals mobilized, or a stable isotope for *in situ* microbial rate studies, or a biocide. The sampler design also needed to consider the possibility of storing the sampler at *in situ* temperatures, either in the hole or on the ship, to conduct incubation and other time-dependent experiments. A large volume of sample (1 L) with an easy-to-access sampling port were desired to aliquot fluids into a range of sample containers for a myriad of chemical and microbial analytical assays. Additional design criteria included (a) diameter, such that the sampler would fit within the confines of the drill pipe used by IODP, (b) modular framework, so that multiple samplers with different treatments could be deployed during a single lowering, (c) compatibility with other borehole instruments, and (d) deployment with a wireline system, using either a drilling vessel, submersible, or ROV. Most importantly, the sampler design must include a temperature-sensitive trigger that is independent of electronics and can operate at temperatures of ~80-180°C and can withstand higher temperatures.

4. Fabrication and Testing

To meet these design criteria we designed and fabricated a modular, 1 L, syringe-style, titanium fluid sampler in which the sample is only in contact with titanium, two high-temperature silicone o-ring seals and a Viton fluoroelastomer gasket (Figure 1). The syringe design affords sterile sample collection at *in situ* pressures and the sample is contained by a custom spring-loaded titanium and Viton check valve. Because the sampler is not gas-tight, pressures within the sampler are the same as that outside the sampler, similar to the Weiss Titanium sampler that has been used for decades to collect hydrothermal fluid at seafloor depths. If dissolved gases remain undersaturated at shipboard temperatures and pressures in the Walden-Weiss and MTFS systems, then the sampler will provide reliable dissolved gas samples, which would be extracted at ~100 kPa. In contrast, if dissolved gases are supersaturated in either the Walden-Weiss or MTFS samplers, then fluid and or gas will leak out of the Viton check valve during recovery.

S~~The s~~ample is drawn slowly into the sample reservoir to prevent degassing, filling the reservoir within ~10 seconds. Upon recovery a sample is withdrawn after detaching the constant-force spring from the piston (Figure 1). Then the titanium plug, which is adjacent to the intake and has a pipe thread, is removed. A titanium tube is then threaded into this opening with the other end of the tube attached to a sample container or collection device (e.g., syringe, filter, bottles). Fluids are expelled by manually applying pressure to the piston, forcing fluid out of the sample chamber through the titanium tube and into the attached sampling apparatus.

Each sample unit is made of 35.5-inch-long (90.2 cm), 3 inch (7.62 cm) inner diameter (I.D.) (schedule 40) grade 2 titanium seamless tubing (Figure 1) with ACME threads to connect units. Connectors were made from 3.75-inch titanium rod with ACME threads. The lower section of the unit provides a cavity for fluid mixing while the sampler descends within the borehole. Fluids from this cavity enter the sampler through a check valve and into the 1-L sample chamber as the piston extends to the base of the trigger platform. This platform acts as a guide for the piston and houses the trigger, which, when activated, releases a 28-pound constant force spring to draw in the sample.

Two types of SMAs were used to trigger the samplers. The first was a commercially available Nitinol material (nickel-titanium SMA), available in spring form and suitable for triggers in the 80°C to 90°C range. These springs are relatively weak, thus they were used in a configuration that mechanically leverages the change in the SMA spring's form to release the constant force spring. Two springs were selected for use and tested at least five times by heating the MTFS module in a water-filled bath. The empirically determined activation temperatures were 80°C and 93°C, respectively, with a relative standard deviation of < 2% ("Spring" in Table 1).

A second type of SMA (CuAlNi) was produced in the shape of a 5 mm diameter rod by TiNi Aerospace. Portions of the rod were cut into 0.6-inch-long pieces and machined to allow a notched titanium bolt to pass. TiNi Aerospace has a proprietary method in which an SMA is heated such that as it reverts back to its original shape, breaking a notched titanium bolt. We tested this trigger process within the MTFS using a heated oil-filled (canola) bath to affect the SMA and release the tension on the constant force spring once the bolt broke (Figure 2). Eight SMA pieces were tested, each at least three times, resulting in a range of release temperatures (~100°C -180°C) with a relative standard deviation of <4% for each of the eight pieces ("Bolt" in Table 1).

Other SMAs pieces from this rod material were compressed within a new piece of precision stainless-steel tubing. A trigger was designed in which the SMA was ejected by a spring once the SMA was warmed to the prescribed temperature. Six SMA pieces from two alloys were tested, each at least three times, resulting in a range of release temperatures (~134°C to 159°C) with a relative standard deviation of <7% for each of the six pieces ("Precision Tube" in Table 1). Unlike the other two triggers, this trigger was tested alone and not within the MTFS.

~~These initial tests were conducted prior to IODP Exp. 385T.~~ Similar to the Walden-Weiss samplers, the MTFS has no implodable volumes and the bulk modulus of titanium requires pressures to deform the material that are well in excess of the deepest of boreholes under hydrostatic pressure. Considering the pressure effect on activation temperature for these SMAs is about 5K/GPa (Kakeshita et al., 1988 and 1999), no pressure tests were conducted on the MTSF triggers. Except for a prototype, which was tested at the seafloor (31 MPa) to assess the piston-syringe mechanism, all systems tests were conducted in water- or oil-filled baths prior to IODP Exp. 385T. We continue to improve the MTFS system. For example, in 2020 the "bolt" trigger option will be re-calibrated using precision torque wrenches that will hopefully improve the repeatability, and lower the standard deviation of the temperature that the trigger activates.

5. Applications

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The first use of the MTFS was planned during the re-entry of DSDP Hole 504B on IODP Exp. 385T "Panama Basin Crustal Architecture and Deep Biosphere: Revisiting Hole 504B and 896A" (Tominaga et al., 2019). The goal was to clear scientific equipment in both holes, sample borehole fluids, and log the boreholes. Neither hole was cleared, thus the MTSF was not deployed (Figure 3). DSDP Hole 504B is ~190°C at the base of the open borehole (Guerin et al., 1996), making it an ideal hole to assess the thermal limits of life in basaltic crust. The current verified thermal limit for life is 122°C (Takai et al., 2008; Clarke, 2014); however, the thermal limit for life may reach or exceed 150°C (Wharton, 2007; Hoehler, 2007), based on (1) protein and lipid structure that compensate for high temperatures, (2) increased stability of ribosomal and transfer RNA at high temperatures, and (3) increased thermal stability of proteins at higher pressures among other arguments (Galtier and Lobry, 1997; Holland and Baross, 2003). The current MTSF design and availability of triggers allows for up to 12 samples to be collected during a single lowering in the interval from 80°C to 181°C.

A second deployment was proposed to collect flocculent material from Ocean Drilling Program (ODP) Hole 896A, which was based on a biofilm-forming microorganisms and images from a downhole camera (Becker et al., 2004; Nigro et al., 2012). This biofilm is distinctly different from those observed within the eastern flank of the Juan de Fuca Ridge, even though thermal and chemical compositions of formation fluids are nearly identical. Differences may suggest site-specific characteristics or biogeographic influences. The MTSF triggers would allow for the collection of samples in the interval from 80 to 90°C, which is the temperature at the base of the borehole.

For both deployments we planned on attaching the elevated temperature borehole sensor (ETBS) tool, which measures borehole temperature and records measurements with electronics that are housed within a vacuum jacket (de Ronde et al., 2019). If a vacuum jacketed system is not available self-contained temperature recorders (i.e., Onset Hobo) can withstand temperatures to ~150°C before likely battery failure, but the data may be recoverable according to suppliers. We have not tested this possibility. Such data coupled with measurements of time and wire deployed would provide a measure of the depth within the borehole that the samplers were triggered.

~~The~~ Although we were unable to deploy the MTFS during IODP Exp. 385T, the MTFS is suitable for use in a range of oceanic or continental boreholes where fluid collection is desired in the temperature range of 80°C to 181°C. As noted above, the syringe-style design of the MTFS allows for a broad range of priming fluids (e.g., acid, biocide, metabolic tracers) to conduct a range of potential experiments. In addition, once recovered the samplers can be placed in oil-filled baths for incubation experiments at *in situ* temperatures. Although not a current capability, the intake could be modified to filter a sample *in situ*. The simplicity of the sampler design affords other potential modifications to accommodate a range of community interests. The MTFS is available for community use. Please contact the first author for interest in using the MTFS.

Data Availability. Data are available in the text. Data from additional testing, specifications, and operational manual will reside with IODP.

Author contributions. CGW and CK led this project. CW tested the bolt and tube triggers. RS and AM designed and tested the spring trigger. TF machined, modified, and troubleshot the

MTFS. TP designed the system for use with IODP. HJ designed triggers and provided machine drawings. All authors contributed to the manuscript.

Competing interests. The authors declare that they have no conflict of interest.

Disclaimer. Any use of trade, firm, or product names is for descriptive purposes only.

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Figures

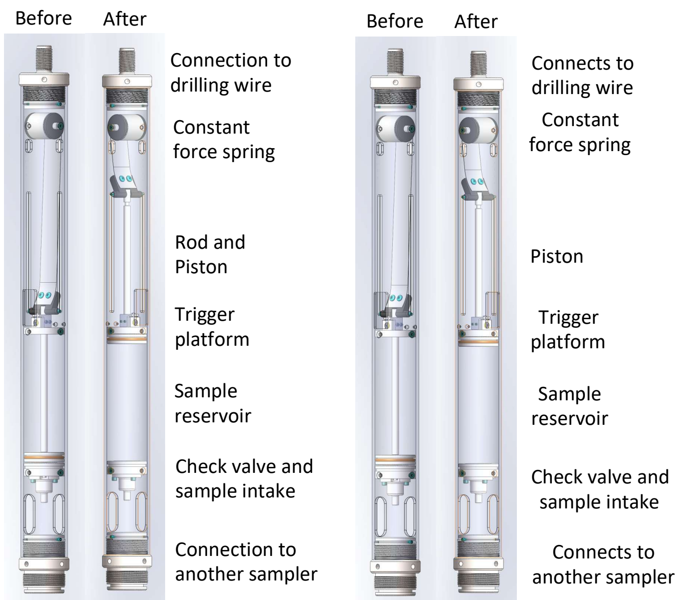

Figure 1. Cross section of a single module of the MTFS highlighting its primary components before the sampler is triggered and after.

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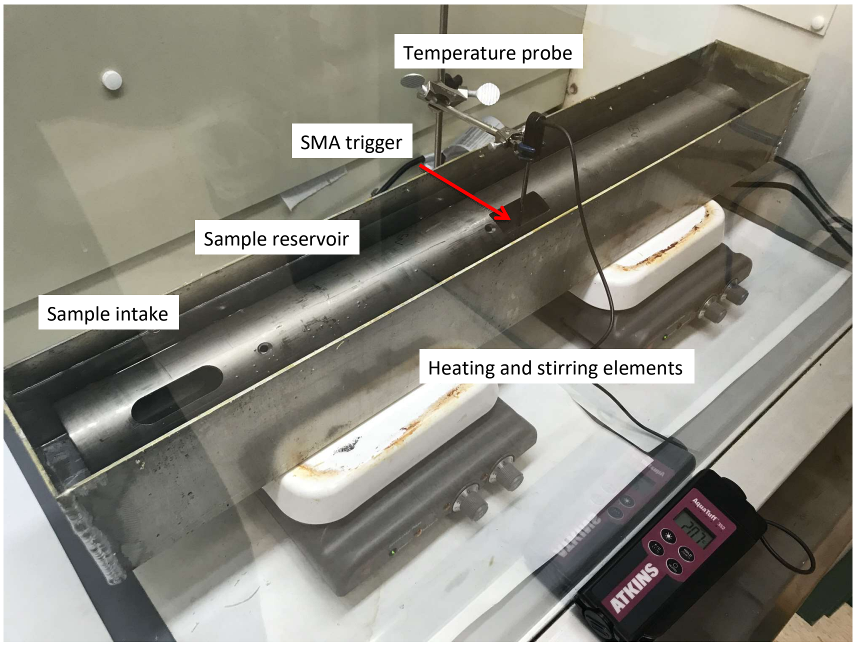

Figure 2. Tests of trigger mechanisms were conducted in a custom bath of canola oil within a chemical hood. The bath holds one 35.5 inch-long (0.90 meter) MTFS module, two magnetic stir bars and a temperature sensor. The temperature sensor is placed within millimeters of the shape metal alloy (SMA) to document the temperature that the trigger is activated. Two heaters with magnetic stirrers keep the oil bath well mixed, heating the bath to 190°C in 1 hour.

Figure 3. Eleven modules were combined in three section on the catwalk in anticipation for deployment within DSDP Hole 504B on IODP Exp. 385T in August 2019 (Tominaga et al., 2019). Final assembly and connection to other sensing instruments would occur on the rig floor, which is up the four steps at the end of the catwalk. Aseptic aluminum foil covers the fluid intake to minimize microbial contamination prior to deployment.

Table 1. Average temperature and standard deviation that an SMA trigger was activated. Data from three trigger mechanisms and multiple discrete SMAs are listed for combinations that were tested more than three times.

Type of Trigger	Temperature and standard deviation (°C)
Spring	80±1; 93±2;
Bolt	102±2; 107±1; 126±3; 128±5; 136±3; 161±4; 174±3; 181±7
Precision Tube	134±9; 152±5; 152±8; 155±7; 157±2; 159±4