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NITINOL TEMPERATURE MONITORING DEVICES

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William J. Buehler, et al

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Naval Surface Weapons Center Silver Spring, Maryland

9 January 1976

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NITINOL TEMPERATURE MONITORING DEVICES

Investigations reported in this document were carried out at the Naval Surface Weapons Center, White Oak under funds from the U. S. Army Medical Research and Development Command, Washington, D. C. 20314, under Task NOL 93030121.

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INTRODUCTION

Early investigations¹⁻⁶ conducted at the U. S. Naval Ordnance Laboratory uncovered a new class of novel non-magnetic nickel-titanium alloys based upon the ductile intermetallic compound TiNi. These alloys were subsequently given the name "Nitinol" which was derived from Ni-Ti-NOL. The latter represents the chemical symbols for the metallic elements nickel and titanium coupled with the abbreviation for the Navai Ordnance Laboratory.

The great interest in the near stoichiometric TiNi composition alloys (55-Nitinol) has stemmed from their unusual mechanical "memory". This thermo-mechanical shape memory allows 55-Nitinol to return to a preset shape after mechanical distortion. Heating above a critical "transition temperature range" (TTR) is the necessary energy input that will induce recovery to the original pre-deformation shape. The critical TTR, over which the material recovers its shape, is primarily a function of alloy composition. This TTR-compositon

- W. J. Buehler and R. C. Wiley, "The Properties of TiNi and Associated Phases", U. S. Naval Ordnance Laboratory NOLTR 61-75, 3 August 1961. Also appeared as U. S. Department Commerce Office of Technical Services Report AD 26607
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- 6. W. J. Buehler and F. E. Wang, "Martensitic Transformations in the TiNi Compound", Proceedings of the 5th International Publishing Company, Aug 1964

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relationship is shown in Figures 1(a) and 1(b)⁷. It may be varied rather precisely over a temperature spectrum from -196°C (liquid nitrogen) to 125°C. However, it should be noted that the recoverable straining must be performed below the TTR of the alloy. Figure 2 illustrates a typical strain-unload-heat recovery cycle. For simplicity this is shown on a stress-strain diagram, where the given tensile loading and unloading is performed below the TTR. In this example (Figure 2), only the heating portion of the cycle given by A' + A", B' + B" and C' + C" exceeds the TTR.

Accompanying the shape recovery is a large energy conversion (heat \rightarrow mechanical) which is capable of overtly exerting large force or "recovery stress". Values of recovery stress in excess of 100,000 psi have been reported⁸ when uniaxial plastic straining of 6 to 8% was used. Figure 3 shows some typical recovery stress curves as a function of strain and temperature.

In addition to the unusual shape memory property the nominal 55-Nitinol alloy exhibits unique changes in yield strength (Y.S) and elastic modulus (E) as a function of heating through the TTR^{8,9}. Yield strength increase of 7:1 and elastic modulus increase of 3:1 with heating through the TTR are typical.

Coupled with the above unique properties are such collateral properties as good corrosion resistance, inherent lower density and being stably non-magnetic.

NITINOL TEMPERATURE MONITOR - MATERIAL CHARACTERISTICS

Previous research and development on the Nitinol material has clearly indicated its potential in the temperature monitoring field. Such criteria as uniformly reliable strain-heat-recovery and composition-related recovery range are basic to temperature monitoring devices. Figure 4 shows a typical heat-induced recovery curve for a bent wire specimen, where the bending was performed below the TTR of the subject alloy. The initial straining (bending) is usually accompanied by some "elastic recovery" (springback). Then with heating the bent wire sample will recover its original straight (memory) configuration. In bending deformation mode, if an outer fiber strain level of 6 to 8% is not exceeded, complete heat-induced recovery should occur over an 8° to 10°C (14° to 18°F) range. That is for the subject alloy once the heat-induced recovery from a bending

^{7.} W. J. Buehler and F. E. Wang, "A Summary of Recent Research on the Nitinol Alloys and Their Potential Applications in Ocean Engineering", Ocean Engineering, vol. 1, pp 105,120, 1968

W. B. Cross, A. H. Kariotis and F. J. Stimler, "Nitinol Characterisation Study", NASA Contract Peport, NASA CR-1433, Sep 1969

^{9.} W. J. Buehler and W. B. Cross, "55-Nitinol -- Unique Wire Alloy with a Memory", Wire Journal, Jun 1969

mode has commenced an 9° to 10°C higher temperature will usually complete the total shape recovery. This range of recovery is also based upon a situation of no constraining load on the bent sample. The basic nature of the transition appears to preclude any narrowing of this temperature recovery range. Conversely, the range is frequently broadened by alloy purity, extent of second phase formation, thermal treatment, etc.

Is the recovery limited to only a bending mode of plastic deformation? The answer to this question is definitely no. A11 plastic deformation modes, e.g., uniaxial tension, compression, torsion and combinations of strain respond equally well to heatinduced recovery. Providing, of course, that the straining is performed below the TTR and does not exceed a critical level-usually 6 to 88⁸. Basic research into the crystallography and mechanism of the strain-heat-recovery^{10,11} reveals the compliant nature of the process. Recovery with heating is always in an equal and opposite direction to the original plastic strain. Further, these basic studies shed light upon the recovery limitation when the plastic straining exceeds about 6 to 8%. Overstraining also causes a drastic reduction in recovery stress (force). Figures 3 and 5, from Cross et al⁸, show the effect of initial scrain on recovery stress. While the strain level varies slightly with deformation mode it appears in Figure 5 to "peak out" in tension near 8%.

No attempt will be made in this report to describe the alloying, mechanical processing and thermal treatment used to produce Nitinol samples with suitable TTR's. This subject area is to be described in a separate report. So for the purposes of this study it will be assumed that optimum sample materials were used.

NITINOL TEMPERATURE MONITOR - DESIGN CONSIDERATIONS

The main thrust of this study was to develop a simple, inexpensive, disposable (or reusable), reliable, fool-proof temperature monitoring device to be used in connection with the storage and/or shipment of whole blood. This device should conclusively reveal whether the blood in question had been excessively warmed in storage or transit, thus rendering it suspect for normal medical use. The authors were informed by medical technicians that the quality and stability of the whole blood was somewhat insured in the temperature range from slightly above 0°C to about 6°C.

11. F. W. Wang, S. J. Pickart and H. A. Alperin, "Mechanism of the TiNi Martensitic Transformation and Crystal Structures of TiNi - II and TiNi - III Phases", J. Applied Physics, vcl. 43 No. 1, Jan 1972, p. 97

^{10.} F. E. Wang, "Transformation Twinning of B2 (CsCl) - Type Structure Based on an Inhomogeneous Shear Model", J. Appl. Phyics, vol. 43, No. 1, Jan 1972, p. 92

Temperatures, above and below this range were to be avoided if possible. Commonly ice water was used as a cooling media and virtually insured the blood against freezing. As a result, the blood monitoring program was to be concerned mainly with excessive heating. Not only was it important to know that the temperature exceeded 6°C, but roughly by how much it exceeded this upper level. Collaterally, it was stated by medical technicians that blood heated in excess of 16 to 18°C for any extended time period rendered the blood quite inferior and suspect for eventual use. Because of the unconstrained recovery temperature range of about 10°C for the Nitinol material, it appeared well suited to monitoring blood overheating in storage or shipment.

With the above general criteria established two immediate steps were taken:

1. Produce a suitable Nitinol-base alloy, in the required wrought form, with a TTR between about 6°C and about 16°C. The basic techniques used to produce controlled TTR alloys are discussed in a separate report¹².

2. Design a device, utilizing the material in (1) above, that would provide a simple, inexpensive, reliable and virtually fool-proof temperature monitoring device.

Once a suitable alloy was produced it remained only to optimize the design of a device. In this connection three principal plastic deformation modes were considered. These were (1) uniaxial tension, (2) torsion or twisting and (3) bending (combined tension and compression stressing). The three are schematically shown in Figure 6. Compression, wnile a very useful mode, was not considered here because of the difficulty and complexity associated with this mode of stressing or straining in practice. It was desirable from a simplicity standpoint to only consider the above three modes, since all could easily be accomplished with a minimum of effort and tools.

Tension, torsion and bending modes were established as the basis for a Nitinol blood temperature monitor. The next step was to design devices that would then satisfy the other basic criteria-simplicity, inexpensive, reliable, disposable, etc.

NITINOL TEMPERATURE MONITOR - DEVICE DESIGNS

Initial efforts were devoted to a "paper" design based upon a Nitinol deformation mode of uniaxial tension. Figure 7 shows such an initial design. In this figure several wires of slightly varying TTR were to be used. All wires were to be positioned in sequence and stretched uniaxially with the aid of a special tool when the entire device had been cooled to a temperature below the wire element with

12. Report in preparation at the Naval Surface Weapons Center, White Oak, by the present author (W. J. Buehler)

lowest TTR. Reliable temperature reading was to be insured by having a spring-loaded rotating indicator which could advance only when a wire contracted sufficiently to go below the "flusn" position. This indicator would allow direct temperature reading based upon the TTR's of the wire elements.

While this device offered a broad temperature monitoring range it had certain obvious shortcomings. The relatively small reversible uniaxial strain capability (6-8%) forced the device to be longer than desired. A second problem was its complexity and construction cost. Lastly, and this objection was really seen later with other designs, where would such a device be positioned to be sensitive to the temperature of the blood within the blood bag? Based upon these objections, and an inability to drastically simplify the lesign, uniaxial tension was abandoned. It was with regret that uniaxial tension had to be abandoned, because of the known accuracy on a cycle-to-cycle basis of this strain-heat-recovery mode. Indicating and switching devices under test, employing tension straining of Nitinol, had been reported¹³ to run in excess of 50.X10⁶ cycles with minimal adjustment.

The next design approach was in the direction of torsion or twisting. This deformation mode was favored over bending, because after tension it had been inherently the most predictable. In this case a "simple" single wire device was proposed. However, as in the uniaxial tension device, the proper amount of straining (twisting) was only possible in a wire of some length, since torsion strain is equal to the wire length divided by the twist angle and is related to the wire radius. Further, in order to accurately set and monitor temperature-recovery relations in torsion required more complexity than desired. This can be seen in the complexity of the device shown in Figures 8 and 9. Based upon variable measurement results and the device complexity this approach was also set aside.

It had been recognized from the outset that the maximum dimensional movement was possible in the minimum size device by employing a sensor of Nitinol that was strained in <u>bending</u>. However, the authors had two serious reservations regarding this Nitinol deformation mode. These were, (1) a fear about the accuracy of the recovery, particularly when used repeatedly, and (2) the design of a temperature monitoring measurement device based upon a bending mode.

Faced with the certain requirement of having to employ a <u>bent</u> Nitinol temperature monitor the authors consulted their sponsor¹⁴ for

^{13.} Private communication with Robert Pike of the Foxboro Company

^{14.} Private communication with Lt. Col. Foster Taft, U. S. Army Medical Research and Development Command

a redefinition of the problem. From this discussion it was reemphasized that blood is satisfactorily stored between about 0°C. 6°C, and that one only need know whether the blood temperature exce 6°C.

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Initially a typical 0.030 inch diameter annealed wire with high transition temperature was studied under bend-heat-recovery conditions. It was found if such a wire was deformed around a mand of suitable diameter to introduce an outer-fiber strain of about 8% the unloaded recovery profile was similar to that shown in sequence Figure 10. Onset of recovery for this wire begins at 54°C and is complete at 64°C. Major recovery occurring between 60° and 62°C. Producing a definite non-linear recovery curve as a function of temperature.

Knowing the basics of the bend recovery process in Nitinol, two monitoring device approaches were conceived and tested. The first based upon the use of a partially annealed cold drawn wire made from an alloy with a TTR near the 6° to 16°C range. The partial annealis of a section of the Nitinol wire was a unique approach designed to provide a bent Nitinol wire that both recovers and simultaneously accurately indicates. Figure 11 cohematically compares the bending characteristics of fully (b) and partially (c) annealed wires. The former bends and recovers nicely but its free-end section is of lit value as a temperature indicator. Conversely, the partially annealed wire (c) will only bend where annealed, and as a result its free-end section has the requirements for serving as an indicator.

It was based on the partial anneal concept that the device sho in Figures 12 and 13 was designed and constructed. Figure 12 shows bent partially annealed wire (white) indicating temperature about mid-range. The ratchet-like rack behind wire indicator was to prev any "relaxation" (or rebending) in the event of cooling after heating This "relaxation" behavior is particularly prevalent in Nitinol elements cycled many times in bending and heat recovery. Figure 13 shows entire monitor assembly. This includes an O-ring that snaps into a suitable groove in the base piece. Then the clear plastic cover with O-ring groove snaps over the base piece and O-ring in bas The clear plastic cover has a " atcher" arm that extends downward. When the cover is rotated on the common O-ring, the arm catches the Nitinol wire and lifts it slightly freeing it from the ratchet and rebends the wire sensor. Of course it should be reemphasized that bending can only occur if the ambient temperature is made lower than the wire TTR. If this condition is met the wire sensor element is bent to the left end of the ratchet, springs back slightly (elastic recovery) and is then ready to again straighten as the temperature rises. Table I presents data obtained from two separate runs. A quick examination of the data reveals the fairly close similarity in bend recovery as a function of temperature for the two runs.

A closer observation of the bend device shown in Figures 12 an 13 reveal certain salient points. The assembly, joined at the O-ri

is essentially water tight. Yet there is freedom to rotate the base and cover at the O-ring allowing resetting (rebending) of the indicator. The entire device could be made inexpensively from two molded plastic pieces, a partially annealed Nikinol wire and an O-ring. The Nitinol wire could be epoxy-cemented in place.

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One limitation of a device of this type is the requirement to place it in close proximity to the blood itself. This can best be accomplished by adding a "kangaroo pouch" on the out-side of the blood bag wall into which the indicator could be placed. Setting the device, or bending the wire, would be done when the blood bag and monitoring device equilibrated to a temperature below the TTR of the Nitinol wire device.

Recognizing the advantages of placing the temperature sensor in intimate contact with the blood a second bend device was conceived. This system would simply employ an annealed Nitinol strip with a nominal 10°C TTR, preferably 6° to 16°C. The strip would be given a U-bend below 6°C and its recovery shape would semi-quantatively allow an estimation of the highest exposure temperature. Such an alloy strip was produced. Figure 14 shows the heat actuated recovery of the Nitinol strip in progressively warmed water. Based upon these sequence photographs it can be seen how a temperature estimate accurate to +2°C could be realized.

How might such a temperature sensing strip be used to monitor whole blood temperatures? The simpliest scheme might be to insert the strip inside the blood bag. Then bend it through the flexible bag wall after the blood is chilled to near 0°C. Figure 15 shows how this was accomplished in a real blood bag filled with water. The bent strip, water and bag were allowed to warm to room temperature. Sequence photographs (b) and (c) show nearly completed and completed recovery, respectively. The entire time span for recovery (a) to (c), in the unagitated state, took about two hours and ten minutes. This time span could be made to vary depending on agitation, strip position in bag, fullness of bag, degrees of supercool below 6°C, etc.

While the simplicity and cost of this system is great a few new potential problem factors arise. These are:

1. Can the metallic Nitinol strip be placed in the blood directly?

2. Will the loose strip create blockage problems?

3. Will bending present some problems?

4. Will location and reading of the strip be difficult in somewhat opaque whole blood?

To address these problems two steps were taker. First the 6° to 18°C Nitinol strip was loosely encased in polyethylene sheaths of two

thicknesses. These bend-recovery data, as a function of time, are given in Table II. Comparison of these data reveal the delay time fo recovery to be about the same for the 0.004 inch and 0.015 inch thick sheaths. Further, the 0.019 inch thick strip produced sufficient straightening force during warming that the presence of the plastic sheath had negligible effect. Based on this study, any problem arising from direct contact between the Nitinol strip and whole blood could be avoided by encasing the strip in a suitable plastic sheath. The other potential problem areas of the loose strip blocking blood flow from the bag and difficulties bending and reading the strip configuration appeared solvable through design.

Figure 16 presents a typical design that is both readily workabl and inexpensive. In this design a plastic assembly consisting of a sealed sheath is bonded at its center to a cylindrical hub. The sheath would encase the Nitinol sensor strip and the hub would be bonded to the <u>inside</u> wall of the blood bag. This entire assembly would be bonded in place before the two bag halves were heat bonded together.

The proposed design should be very utilitarian. That is the hub would serve both as an anchor for the Nitinol monitor strip and its diameter would provide an optimum mandrel around which to bend the strip. Further, the encased strip could be readily bent and manipulated through the bag wall. Finally, the mechanics of observin the contour of the monitor strip would be very simple due to the closeness of its profile to the transparent bag wall.

Such a device as shown in Figure 16 probably represents the ultimate in monitoring efficiency. It is continuously in a bath of blood that in transit would be agitated sufficiently to promote a uniform temperature in the blood. Yet a few thousands of an inch of inert plastic provides complete assurance against any possible metal-blood interaction that could result in contamination of the blood.

SUMMARY

Several schemes for monitoring blood temperature were examined. Tension and torsion deformation modes were rejected in favor of bending. The latter bend-heat-recovery sequence provided the most efficient, simpliest and least expensive monitor.

Based upon the above information many obvious design variations are possible. This report is mainly intended to show the feasibility of the Nitinol material as a temperature monitor. Further, the wide range of TTR's possible in the Nitinol materials through alloying provides monitoring possibilities in many temperature ranges and applications.

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The authors wish to acknowledge the U. S. Army Medical Research and Development Command for sponsoring this study.

TABLES 1 MONITOR INDICATOR POSITION FOR VARIOUS TEMPERATURES

Two bend-heat-recovery runs made on the "partially annealed" Nitinol wire indicator

RUN #1

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RUN #2

$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Indicator Position	Temperature (°C)	
10.4 11.9 11 12 11 12.1 12.5 12.5	0-4Elastic Recovery 4 4.3 4.8 5.2 5.8 6.3 6.5 7 8 9.2 9.5 9.7 9.9 10 10.2 10.4 11 11 11 12.5	3 3 5 7 8 8.5 9 9.3 9.3 9.3 9.3 9.3 10.2 10.2 10.2 10.5 10.9 11.1 11.2 11.3 11.5 11.8 11.9 12.1 12.5	

Indicator Position	Temperature (^O C)
0-4 Elastic	3
Recovery	
4.1	3.5
4.4	5.2
4.5	5.3
4.C 5	62
5.2	6.8
5.3	7
5.3	7.5
5.4	7.6
5.0	8
5.0 5.0	0.1 8.2
6.1	8.8
6.3	9
6.5	9.2
7.2	9.8
(•5	10
8.5	.10.1
9	10.5
9.1	10.8
9.2	10.9
9.3	
9.2	11.5
10	11.8
10	12
12	12.2
12.5	12.5
12.0 74	15 14 R
15	17
16.7	20.1
	{

YABLE II

Data pertaining to the time of the heat actuated recovery of a U-Bent 0.019 inch thick Nitinol strip (6 to 18 C transition temperature range) encased in polyethylene sheaths*



SHEATH THICK. = 0.004 inch.

Bend Angle	Time
(Degrees)	(minutes)
65 76 100 125 150 165 175 180	0 0.5 1.0 1.5 2.0 2.5 3.0 3.5

SHEATH THICK. = 0.015 incl.

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Bend Angle	Time
(degrees)	(minutes
55 65 90 117 135 151 165 174 180	0 0.50 1.50 2.50 3.50 3.50 3.4

*Recovery heating was done in still air



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CALLENS!

Shows the wide variation in the martensitic transition temperature (MAS) possible by substituting cobalt for nickel.



b Martensitic transition temperature (A_s) change with minor variations in the Ti:Ni ratio. Note the maximum occurs close to 55-Nitinol composition (stoichiometric TiNi).

FIG. 1 (a), (b) MARTENSITIU TRANSITION TEMPERATURES AS A FUNCTION OF COMPOSITION

NSWC/WOL/TR 75-140



STRAIN

Typical stress-strain curve for nominal 55-Nitinol when tested below the TTR. Cycles A-A'-A'' and B-B'-B'' show the strain-heat-recovery of 55-Nitinol when strained below the critical level. Cycle C-C'-C''shows the result of over-straining on heat actuated recovery.





Typical recovery stress curves for Nitinol wire when strained in tension to various levels and heated.

FIG. 3 RECOVERY STRESS PERFORMANCE OF NITINOL STRAINED TO VARIOUS LEVELS

Typical recovery curve for 55-Nitinol wire bent around a mandrel and then restored to its straight memory configuration by heating (from Batuelle)



FIG. 4 STRAIN RECOVERY OF A BENT NITINOL WIRE



Maximum recovery stress as a function of initial strain (tension). Note the general tendency toward reduced recovery stress (force) when straining exceeds about $8\%^{(8)}$.

FIG. 5 MAXIMUM RECOVERY STRESS AS A FUNCTION OF INITIAL STRAIN



Depicts typical strain-heat-recovery patterns for Nitinol material given three different modes of deformation below the TTR.

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FIG. 6 TYPICAL STRAIN-HEAT-RECOVERY PATTERNS FOR DIFFERENT DEFORMATION MODES

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Tez Spring Actuated Arm ·Ter

 $T_{c1} < T_{c2} < T_{c3}$ ---Etc.

Proposed early design of Nitinol blood temperature monitor. Wires were to be of varied composition and varied TTR. All wires were to be uniaxially strained when the temperature of the wire of lowest TTR had been reached. The spring-loaded rotary lever would be allowed to progress as recovery (contraction) in each composition wire occurred. The rotary indicator would be stopped by uncontracted wire(s) - or those with TTR's higher than the highest exposure temperature.

F/G. 7 EARLY DESIGN OF NITINOL BLOOD TEMPERATURE MONITOR



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FIG. 11 BEND BEHAVIOR OF COMPLETELY ANNEALED WIRE VS THAT OF COLD DRAWN WIRE WITH AN ANNEALED SEGMENT

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with a TTR of 6°C to 18°C. Photographs were taken from directly above while strip was allowed to recover in progressively warmer water in an Sequence photographs of the recovery of a bent Nitinol strip open round glass dish.

FIG. 14 SEQUENCE PHOTOGRAPHS OF RECOVERY OF A BENT NITINOL. STRIP FROM 6° TO 18° C





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FIG. 15 SEQUENCE PHOTOGRAPHS OF RECOVERY OF IN-SITU BENT NITINOL STRIP IN BLOOD BAG

