

First Progress Report
Research and Development on Titanium
Alloys
AF 33(038)-3736
August 31, 1949

Covering the period May 18, 1949
to
September 18, 1949

A Brief Scientific Analysis

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INTRODUCTION

This document was prepared by SUNRISE after receiving a digitised copy of the First Progress Report from Mr Anthony Braglia on March 3, 2010. It is a brief analysis of the official and formerly classified Battelle Memorial Institute's First Progress Report dated August 31, 1949 written under contract¹ for the USAF through AF 33(038)-3736¹. It was released under US FoIA on February 11, 2010 after a request was put through to the Defense Technical Information Center (DTIC) by American researcher Mr Billy Cox.

The aim of this analysis is to understand the work carried out by USAF/Battelle on titanium and its alloys with particular emphasis on the world's lightest and most powerful titanium-base shape memory alloy NiTi and to help determine whether there is likely to be any relationship between this work and the dark-greyish shape memory metallic foil found near Roswell and analysed at Wright-Patterson AFB in early July 1947.

The existence of this Second Progress Report was mentioned by an Australian researcher and science graduate from the ANU in the early 1990s. Thinking it might be relevant to the Roswell case, the existence of the report was first mentioned by SUNRISE in Australia between 1998-99 titled *Roswell Revisited: an Australian researcher's view on the famous UFO crash of 1947*:

In 2009, authors Jim Carey and Donald Schmidt re-published the NiTi-Roswell results in their book *Witness to Roswell* in the final chapter written by American researcher Mr Braglia (without providing written acknowledgment of the Australian researcher's contribution).

This document was originally prepared by SUNRISE on March 6, 2010.

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January 28, 2012

¹ There is only one contract number that commenced earlier in relationship to the study of titanium by the USAF and Battelle and that's W 33-038 ac-21229, commencing 18 May 1948.

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1

A General Look at the First Progress Report

- 1.1 The report mentions NiTi (the world's most powerful and lightest titanium-base shape memory alloy of dark-greyish appearance).
- 1.2 Data on NiTi is stated by L.W. Eastwood in his letter dated 16 September 1949 as officially published in the last report of Contract Number W 33-038 ac-21229, which is said to be a Summary Report (either Part I, II or III) (PDF p.7).
- 1.3 This report discusses two binary alloy called TiNi and TiGe possibly for comparison purposes.
- 1.4 Two ternary titanium alloys comprising molybdenum and manganese combined with a selection of elements such as nitrogen, copper, chromium, iron, cobalt and carbon were studied.
- 1.5 The alloys developed for this report were tested in "both the as-hot-rolled temper and after aging the hot-rolled sheet four hours at 750°F". Tensile strengths, hardness and elongations were measured.
- 1.6 The report also looked at materials for building crucibles for holding the molten titanium metal and alloys.
- 1.7 Mechanical properties such as tensile strength, hardness and elongation for NiTi are included in this report.
- 1.8 A significant amount of work is made in this report to understand how to measure and minimise oxygen impurities entering titanium metal and its alloys. This work is carried out after 18 May 1949. No indications the work occurred earlier than this.



- 1.9 No other known shape memory alloys are mentioned in this report.
- 1.10 The principal Battelle authors for the study of NiTi of Eastwood, Fawn and Craighead claim further study of NiTi is not recommended, but has continued the work into the alloy in the Second Progress Report of this contract number as well as the last report of the previous contract number.
- 1.11 NiTi remained of interest to USAF in 1949.



2

Cover Pages and Letter from L.W. Eastwood, PDF pp.5-7

- 2.1 A formerly restricted report available to Department of Defense (DoD) employees². PDF p.1
- 2.2 Discusses the development of, and data obtained for, selected titanium-base alloys of interest to the USAF at the time. PDF p.4
- 2.3 Work for this report was conducted during the period from 18 May to 18 September 1949. PDF pp.4-6
- 2.4 Battelle scientific authors are stated on the covers as “Simmons, O.W.; Greenidge, C.T.; Craighead, C.M; and others”. The Air Material Command at Wright-Patterson Air Force Base (WPAFB)³ is also stipulated as co-author of the report. PDF p.4.
- 2.5 Mr J. B. Johnson, chief of Materials Laboratory at Wright-Patterson AFB, is mentioned by L.W. Eastwood in his letter dated 16 September 1949 as the principal person of contact for the USAF in relation to all titanium research work including NiTi performed at Battelle.⁴
- 2.6 Eastwood indicates the alloys studied in this report and the data collected therein are also included in the Summary Report of Contract Number W 33-038 ac-21229. As stated: “Data obtained during this period on alloys already under study on May 18, 1949, are included in the above-mentioned Summary Report.”

² Approval from DoD would have been required for outside scientists to view the report unless a copy was held at Battelle and certain individuals had close contact with the authors.

³ Wright-Patterson AFB is the official location for all Roswell debris for analysis.

⁴ Mr J. B. Johnson was also known from a 1965 metals article to have monitored the work of NYU on another shape memory alloy called TiZr.



This suggests interest in NiTi went officially back to Summary Report (either Part I, II or III) of the previous contract.

- 2.7 Eastwood mentioned the importance of oxygen impurities in titanium and were developing analytical methods to measure and find ways to deal with this.
- 2.8 Eastwood also indicated that USAF and Battelle were not sure of the right materials needed to hold molten titanium to make titanium-base alloys after 18 May 1949. They were looking at the “development of refractories for holding molten titanium”.
- 2.9 L. W. Eastwood presented 25 copies of the report to unnamed male individuals at Wright-Patterson AFB through Building 258 and one reproducible copy on 16 September 1949.



3

Table of Contents, PDF p.8

- 3.1 This report would appear to contain no missing pages and there are no additional parts.
- 3.2 The report has 59 pages.
- 3.3 No mention of NiTi in the contents page. Closer analysis of the report shows the alloy was studied.



4

Summary Section

- 4.1 First paragraph mentions the binary alloys of titanium-germanium (TiGe) and titanium-nickel (TiNi).
- 4.2 Ternary titanium-base alloys of interest would contain manganese and molybdenum. About 5 per cent molybdenum was added together with 1 to 2 per cent copper, manganese or iron (p.1; PDF p.9). And about 3.5 to 5 per cent manganese was added together with 0.1 to 2 per cent nitrogen or cobalt (p.2, PDF p.10).
- 4.3 Alloys were fabricated into sheet form (p.2, PDF p.10).
- 4.4 Aging the hot-rolled alloy sheets at 750°F helped to increase tensile strength and hardness of some ternary titanium alloys. Less successful was the solution heat treatment at 1600°F. (p.2, PDF p.10)
- 4.5 Refractories for holding molten metal involved tungsten carbide, titanium carbide, and zirconium carbide. Also attempted to melt titanium in graphite crucibles “lined with tantalum carbide and tungsten boride” but tests were incomplete. There is a concern about temperature of the molten titanium and possibly preventing impurities entering the titanium alloys from the crucible materials used. (p.3, PDF p.11)
- 4.6 Considerable difficulties in getting consistent results when measuring the oxygen in titanium held in the tetrachloride solution at certain temperatures. Main problem was the extra oxygen from the air entering the tests to ruin the results. Battelle investigated the option of doing the measuring inside a vacuum chamber through the vacuum fusion technique and the vacuum-fusion



mass-spectrograph method. Neither approach yielded consistent results. (p.3, PDF p.11)

- 4.7 Wright-Patterson AFB were looking to produce the purest form of titanium possible and looked for methods to measure and reduce the amount of oxygen entering the metal and titanium alloys.



4.8

5

Section titled Arc Melting Titanium-base Alloys, by C.W. Simmons and C.T. Greenidge

- 5.1 The authors indicated “approximately one hundred and forty 0.5-pound ingots were made and submitted for fabrication during the period May 18 to August 31 [1949]”. (p.3, PDF p.11)
- 5.2 “Process A” titanium is the purest form of the metal produced by or is available to Battelle. Mr J. B. Johnson at Wright-Patterson AFB made a request for this Process A metal to be produced in two ingots described as Ti354 and Ti363. Battelle produced 11 pounds gross of Ti354 and 8.5 pounds of Ti363 and “were scalped to 6 and 5.1 pounds, respectively”. These ingots were shipped to Mr E.A. Gee of E. I. du Pont de Nemours and Company. (p.4, PDF p.12)



6

Section titled Evaluation of Experimental Titanium-base Alloys, by C.M. Craighead, F. Fawn and L.W. Eastwood

- 6.1 Authors suggest NiTi and TiGe were hot-rolled and aged for 4 hours to reveal any changes to hardness and strength. (p.4, PDF p.12)
- 6.2 A selection of different compositions of the elements nickel and germanium were added to titanium to help determine changes in hardness and tensile strength.
- 6.3 Germanium was added to titanium in the range 0.01 to 1.0 per cent. Nickel was added to titanium in the range 1.75 to 15.0 per cent (p.6, PDF p.14).
- 6.4 Table 1 suggests the USAF could be seeking a pattern in the tensile strength, elongation and hardness data as more nickel is added to the titanium in NiTi (p.6, PDF p.14). This may be used to give an estimate of the tensile strength in the 50 percent nickel range of the original shape memory NiTi alloy.
- 6.5 The tensile strength pattern indicates an increase in the strength from 80,000 pounds per square inch (psi) but levelling off slightly above 120,000 psi with increasing nickel (p.9, PDF p.17).
- 6.6 NiTi has much higher tensile strength results than TiGe. TiGe showed no improvement to the mechanical properties by adding extra germanium as well as aging the heat rolled alloy at 750°F.
- 6.7 There is also a trend towards increasing hardness under the Vickers Hardness (VHW) column for NiTi and TiGe. There is no levelling off of the hardness



- for NiTi — it simply goes up with increasing percentage of the nickel (p.9, PDF p.17).
- 6.8 The elongation in 1 inch of the samples also shows a pattern with significant changes taking place from 1.75 to 15.0 per cent nickel in NiTi with the percentage decreasing from 22.5 per cent to 2.0 per cent respectively. NOTE: The shape memory effect of NiTi when it goes through its martensitic transformation to a compact crystalline structure is known to reduce its size (ie. affect elongation). (p.6, PDF p.14)
- 6.9 No obvious improvement to tensile strength and hardness of NiTi and TiGe when aged for 4 hours at 750°F after hot-rolled at 1450°F.
- 6.10 Table 2 attempts to measure changes to hardness of NiTi and TiGe when hot rolled at different temperatures and composition of the nickel or germanium elements. Again the results appear inconclusive at different temperatures — only the composition range of the elements added to Process A titanium affects hardness.
- 6.11 The above results suggest the USAF were unaware of increasing tensile strength and hardness through cold-working of the NiTi alloy below 500°C.
- 6.12 While the USAF were interested in the mechanical properties in NiTi in the low Ni composition range to help make extrapolations to a higher Ni composition range, we see that in order to avoid any further interest in these alloys by other scientists, the authors wrote “The present data do not justify further investigation of binary titanium-germanium or titanium-nickel alloys” (p.5, PDF p.13). Yet something made the USAF ask Battelle to study NiTi in the previous report, and would do so again for the next progress report. It is almost as if TiGe was thrown in to make any secret work on NiTi at Wright-Patterson AFB look mundane.
- 6.13 Table 3 (pp.10-12, PDF pp.18-20) looks at the tensile strengths and hardness for the ternary titanium-base alloys of interest in this report. These may be used as further comparisons with NiTi for any secret study on the alloy at Wright-Patterson AFB.



- 6.14 Figures 3-9 (pp.12-18, PDF pp.24-30) indicates greater hardness and tensile strength for the titanium-molybdenum (Ti-Mo) binary alloy compared to NiTi. Adding small amounts of carbon, copper, iron, cobalt, nickel or chromium to TiMo can further improve these mechanical properties either to a minor or more significant degree depending on composition range of these minor elements.
- 6.15 A similar result can be found when adding 0.1 per cent nitrogen to the titanium-manganese alloy without sacrificing the elongation of the alloy. But ductility of the alloy goes down dramatically when 0.2 per cent nitrogen is added. (p.20, PDF p.32)
- 6.16 The “as-hot-rolled temper” gives better tensile strength and hardness results for ternary titanium-manganese base alloys compared to “a solution heat treatment at 1600°F”. However the aging method shows inconsistent results (pp.27-28, PDF pp.41-42)



7

Section titled Investigation of Refractories for Melting Titanium, by P.D. Maddex and L.W. Eastwood

- 7.1 The authors stated work into “unusual refractories” considered necessary for holding molten titanium and its alloys had continued from the previous contract number. (p.28, PDF p.42)
- 7.2 Nearly all previous tests for suitable refractories (or crucibles) were made in a vacuum.
- 7.3 Method of testing involved melting 10 to 15 grams of titanium in small crucibles “using a standardised melting temperature of 3100°F”. (p.28, PDF p.42)
- 7.4 Problems in the test crucible melting have been noticed in the vacuum method. The authors used a purified argon atmosphere instead of a vacuum to eliminate the difficulty.
- 7.5 The authors suggested in Table 7 that “none of the crucible materials, on which tests have been completed, can be considered satisfactory”. (p.29, PDF p.43)
- 7.6 Some sticking of the titanium melt to the sides of the tungsten carbide crucible was noticed. Initially thought to be a porosity difference between the bottom and sides of the crucible, the authors have come to the conclusion that it was probably caused by small temperature differences between the bottom and sides of the crucible. (p.29, PDF p.43)
- 7.7 Negotiations have been made to get the Norton Company “to supply hot pressed crucibles for these studies”.



8

Section titled Analytical Methods for Titanium-base Alloys: The Determination of Oxygen in Titanium by the $\text{Cl}_2 - \text{CCl}_4$ Method, by E.J. Center and A.C. Eckert

- 8.1 Authors indicated the work in this field continued in the last report suggesting Summary Report Part I, II or III or the last bimonthly report of the previous contract number would contain further information. This also suggests the earliest date for all work in this field involving purity issues of titanium by Battelle on behalf of the USAF is no earlier than 18 May 1948 (when the previous contract began).
- 8.2 The authors were interested in any form of decomposition of the titanium tetrachloride method at different temperatures by adding a known amount of titanium oxide (TiO_2) or oxygen gas and let any reaction take place for a fixed amount of time (eg, 2 hours). Then a re-measure of the remaining oxygen may reveal differences. The main concern is how much oxygen can get into the titanium tetrachloride sample.
- 8.3 The authors note decomposition was too low to be measured when the reaction temperature is 400 to 550°C. Temperatures above 600°C were not recommended due to damage to the quartz vacuum tube used in the tests with the samples. (p.31, PDF p.45)
- 8.4 The authors used a higher temperature resistant quartz vacuum tube to conduct the tests. (p.31, PDF p.45)



- 8.5 However considerable “attack” of the quartz tube would still take place at temperatures from 700 to 900°C with decomposition sufficiently complete at 900°C. (p.32, PDF p.46)
- 8.6 The authors found it difficult to measure oxygen impurities using the method outlined in this section.

The Analysis of Titanium for Oxygen by Vacuum-Fusion Methods

By M. W. Mallett, D. G. Thomas, and C.B. Griffith

- 8.7 The authors noted three different modifications were made to the vacuum-fusion method by several laboratories resulting in successful analysis of oxygen in titanium. This section explores these three modification types with a view to a possible improved version when all interfering elements in the apparatus design are eliminated. (p.33, PDF p.47)
- 8.8 In fact, the authors developed another method by adding isotope O^{18} . As the authors stated: “The procedure consisted of diluting a known amount of O^{18} tracer with the unknown amount of oxygen in the titanium. By extracting the gas from the titanium and determining the new ratio of O^{18} to O^{16} with the mass spectrometer, the unknown amount of oxygen can easily be calculated.” (pp.33-34, PDF pp.47-48)
- 8.9 The authors made comparison of their method with the three modifications of the vacuum-fusion method to see which one yielded better and more consistent results.
- 8.10 The authors made it clear that Battelle had developed a method of measuring oxygen in thorium several years ago (p.35, PDF p.49). But no indications it had done the same with titanium until this report and possibly the previous report of the previous contract number. And even in this report, Battelle and the USAF were not satisfied by the purity of the titanium tetrachloride method for some reason. As the first effort into titanium research for the USAF by Battelle commenced on 18 May 1948, there is no evidence to suggest such



work on the problems of getting titanium into the purest form was ever officially discussed with Battelle anytime in 1947.

- 8.11 All methods of generating a vacuum in previous modifications of the vacuum-fusion method were described as slow. More work was being done to improve on this aspect. (p.42, PDF p.57)
- 8.12 Details given by the authors of how they achieved their preferred method of measuring oxygen in titanium using the isotope O^{18} .

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INTRODUCTION

This document was prepared by SUNRISE after receiving a digitised copy of the Second Progress Report from a European researcher in August 2009. It is a brief analysis of the official and formerly classified Battelle Memorial Institute's Second Progress Report dated October 31, 1949 written under contract for the USAF through AF 33(038)-3736⁵. It was released under US FoIA on August 1, 2009 after a request was put through to the Defense Technical Information Center (DTIC) by the European researcher and an American researcher named Mr Billy Cox.

The aim of this analysis is to understand the work carried out by USAF/Battelle on titanium and its alloys with particular emphasis on the world's lightest and most powerful titanium-base shape memory alloy NiTi and to help determine whether there is likely to be any relationship between this work and the dark-greyish shape memory metallic foil found near Roswell and analysed at Wright-Patterson AFB in early July 1947.

The existence of this Second Progress Report was mentioned by an Australian researcher and science graduate from the ANU in the early 1990s. Thinking it might be relevant to the Roswell case, the existence of the report was first mentioned by SUNRISE in Australia between 1998-99 titled *Roswell Revisited: an Australian researcher's view on the famous UFO crash of 1947*:

In 2009, authors Jim Carey and Donald Schmidt re-published the NiTi-Roswell results in their book *Witness to Roswell* in the final chapter written by American researcher Mr Bragalia (without providing written acknowledgment of the Australian researcher's contribution).

This document was originally prepared by SUNRISE on August 28, 2009.

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⁵ There is only one contract number that commenced earlier in relationship to the study of titanium by the USAF and Battelle and that's W 33-038 ac-21229, commencing 18 May 1948.

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1

A General Look at the Second Progress Report

- 1.12 The report mentions NiTi (the world's most powerful and lightest titanium-base shape memory alloy of dark-greyish appearance) and TiZr (another known shape memory alloy considered the cousin to NiTi in terms of the similar chemical properties of Zr and Ti). It would therefore seem reasonable to assume that this is the official Second Progress Report mentioned in the footnotes of several metal articles on NiTi, TiZr and ternary alloys such as NiTiCo and others in the NiTi family under the authors "Eastwood, Fawn and Craighead".
- 1.13 Further details on NiTi exist in the previous bimonthly report of July-August 1949 (p.65, PDF p.15).
- 1.14 The report focuses on (i) establishing the extent of the phase field of the alpha and beta crystalline structures simultaneously present within selected titanium-base alloys including NiTi primarily in the titanium-rich (or low nickel) range and with tentative phase diagrams; (ii) testing for levels of oxygen impurities in standard titanium iodide solutions by adding TiO₂; (iii) measuring mechanical properties (eg. tensile strength and hardness); and (iv) finding suitable materials for making crucibles to hold molten titanium and titanium alloys.
- 1.15 The report may suggest Battelle and USAF were not studying shape memory alloys by focussing attention of the work towards the titanium-rich end of the composition range as well as not using the technical term martensite transformations or pseudoelastic alloys. However the interest in identifying the dual crystalline structures within a phase field of various selected alloys is



considered critical scientific knowledge when understanding shape memory behaviours.

- 1.16 Since the presence of dual crystalline structures has the potential for titanium-base alloys to show evidence of shape memory behaviours, the report mentions at least two notable shape memory alloys of NiTi and TiZr. If the missing pages were supplied, NiTiCo would be another. And there is the potential for NiTiCr to be a shape memory alloy as well (to be confirmed later).
- 1.17 This report does not include data from the previous bimonthly report on mechanical properties for NiTi (which we must assume contains tensile strength and hardness data).
- 1.18 The report suggests the USAF were looking for an alternative titanium-base alloy of similar hardness and tensile strength that wouldn't involve the use of nickel-titanium. The most promising candidate is one involving the addition of vanadium to titanium so long as the element is properly homogenised throughout (p.92, PDF p.44).



2

Cover Pages, PDF pp.1, 6 & 7

- 2.10 A formerly restricted report available to Department of Defense (DoD) employees⁶. PDF p.5
- 2.11 Discusses the development of selected titanium-base alloys and data obtained of interest to the USAF at the time.
- 2.12 Work was conducted during the period from 1 September to 31 October 1949. PDF p.5
- 2.13 Battelle scientific authors are stated on the covers as “Simmons, O.W.; Greenidge, C.T.; Craighead, C.M; and others”. The Air Material Command at Wright-Patterson Air Force Base (WPAFB)⁷ is also stipulated as co-author of the report. PDF p.5.⁸
- 2.14 L. W. Eastwood presented 30 copies of the report to unnamed male individuals at Wright-Patterson AFB through Building 258 and one reproducible copy on 11 November 1949.

⁶ Approval from DoD would have been required for outside scientists to view the report unless a copy was held at Battelle and certain individuals had close contact with the authors.

⁷ Wright-Patterson AFB is the official location for all Roswell debris for analysis.

⁸ Metal journals mentioning this report in the footnotes claim it is Craighead, Eastwood and Fawn. There must be a specific section in the report mentioning these three authors.



3

Table of Contents, pp.58-59 or PDF pp.8-9

- 3.4 The report has missing pages.
- 3.5 The missing pages should contain TiNiCo (a shape memory alloy) according to footnote number 99 in H.O. Teeple's *Nickel and High-Nickel Alloys* (Industrial and Engineering Chemistry, October 1950, Volume 42 Number 10, pp.1990-2001) As Teeple (from The International Nickel Company, Inc. in New York) stated on page 1990:

“Craighead et al. (99) reported binary alloys of titanium with nickel and cobalt and ternary alloys of titanium-chromium alloys with nickel and cobalt among others. Charts on properties and diagrams are included.”

This quote claims other alloys not mentioned in this report are TiNiCo, TiNiCr and TiCrCo.

- 3.6 It would appear the Second Progress Report is divided into two parts: pages 1 to 59 (missing pages) and pages 60 to 119 (released under US FoIA).
- 3.7 No mention of the titanium-nickel alloy in the contents page. Yet closer analysis of the report clearly shows the alloy was studied.⁹

⁹ How did it come to the attention of several metal journals over the existence of the NiTi study in this restricted USAF-Battelle report? Someone must have seen the original report and mentioned it in a footnote. It is now starting to look like it began through Teeple in 1950. His work on nickel and paper manufacturing as early as 1946 had given him contacts at Battelle where he learned about the work of Craighead, Fawn and Eastwood on the nickel-titanium alloy and ternary alloy versions of it and mentioned it in his article.



4

Summary Section

- 4.9 First paragraph mentions the binary alloys of titanium-germanium (TiGe) and titanium-nickel (TiNi).
- 4.10 The amount of germanium and beryllium added in the binary alloys is between 0 and 1 per cent (p.60, PDF p.10) and 0.1 to 1.0 per cent (p.60, PDF p.10) respectively. The addition of nickel to titanium to form NiTi is in the range 0-10% nickel.
- 4.11 Additions of 1 and 2 per cent columbium and tantalum were added to “Process A” titanium (p.61, PDF p.11).
- 4.12 The composition range of interest in this report for the nickel-titanium alloy was also applied to the zirconium-titanium system (ie. 0 to 10 per cent zirconium).¹⁰
- 4.13 Other binary alloys studied include silver-titanium (p.60, PDF p.10), titanium-beryllium (p.60, PDF p.10), titanium-zirconium (p.60, PDF p.10), titanium-tantalum (p.61, PDF p.11), and titanium-columbium (p.61, PDF p.11).
- 4.14 Ternary titanium-base alloys include manganese and vanadium, and molybdenum and tungsten (p.61; PDF p.11).
- 4.15 Work into titanium-base binary alloys involved determining the extent of the dual crystalline phase field (ie. containing alpha and beta structures), and the mechanical properties of tensile strength and hardness “after fabrication to sheet” form (pp.60-61, PDF pp.10-11)

¹⁰ This suggests Battelle and USAF were trying to compare TiNi and TiZr and make sheet samples so they could secretly test for shape memory effects. Perhaps it is at this point that something was discovered in TiZr?



- 4.16 “Fabrication to sheet” form for the alloys was integral when testing for tensile properties such as hardness (p.61, PDF p.11).¹¹
- 4.17 There is discussion of “refractories for holding molten titanium” (p.61, PDF p.11) and measuring the oxygen impurities in titanium using “the vacuum-fusion technique” (p.62, PDF p.12).

¹¹ The Roswell metallic foil was fabricated in sheet form.



5

Section titled Arc Melting Titanium-base Alloys, by C.W. Simmons and C.T. Greenidge

- 5.3 The authors indicated “approximately thirty-five 0.5-pound ingots were made and submitted for fabrication during the period September 1 to October 15, 1949”.
- 5.4 “Process A” metal appears to be the purest form of titanium produced by or is available to Battelle. The process probably involved using titanium iodide or titanium tetrachloride where the metal was later chemically separated and heated through arc melting within small crucibles inside a vacuum furnace to help produce pure titanium and to combine other elements to the metal to form high quality alloys.
- 5.5 A problem was noted in combining manganese with titanium due to the low density of manganese and how it tends to separate from the molten solution. The authors suggested (i) adding a master alloy; (ii) “arc melting electrolytic manganese and Process A titanium”; (iii) crushing the alloy to a small size; (iv) compressing into bars; and (v) sintering for a period of time at high temperatures to form the final ingots for analysis (p.63, PDF p.13).



6

Section titled Evaluation of Experimental Titanium-base Alloys, by C.M. Craighead, F. Fawn and L.W. Eastwood

- 6.17 This is the official section of the report referred to in the footnotes of several metal articles.
- 6.18 First half of the work involves identifying the extent of the dual crystalline beta and alpha phase fields at the titanium-rich end of the composition of several binary titanium-base alloys and in determining the solvus lines (the boundary between the phase fields). Rapid quenching in cold water of the alloys at different compositions and temperatures is used to lock in the crystalline structures (p.65, PDF p.15).¹²
- 6.19 Among the results gathered is how adding a little germanium to titanium would “raise the beta and lower the alpha solvus lines” (p.65, PDF p.15).
- 6.20 Adding nickel to titanium to form NiTi “was found to limit markedly the alpha-phase field and to lower the beta solvus line” (p.68, PDF p.18).
- 6.21 According to the report: “A nickel-rich phase appeared in the microstructure of the [NiTi] alloy containing about 7.5 per cent nickel when the specimen was quenched from 1450°F, but is absent...in the specimen quenched from 1550°F. From the available data, the eutectoid composition [in the binary titanium-nickel system] is placed between 6 and 7 per cent nickel.” (p.68, PDF p.18)

¹²

A solid-to-solid crystalline change from one type to another when heated is currently the leading explanation for how the shape memory effect works in alloys of this type.



- 6.22 A nickel-rich (gamma) phase means the nickel atoms are able to cluster or stick together somewhat within the titanium matrix structure. Higher temperatures help to eliminate the gamma phase.
- 6.23 The report mentions the “additions of 1 to 10 per cent zirconium were also studied” (p.60, PDF p.10) or “Additions of 1.0, 2.5, 3.5, 5.0 and 10.0 per cent zirconium were made to Process A titanium” (p.80, PDF p.32) but claim “No alloys of interest were noted” (p.60, PDF p.10).¹³
- 6.24 We also find a mention of additional information on the nickel-titanium alloy available in the previous bimonthly report claiming “data on the mechanical properties and the response to heat treatment and aging” is revealed in Table 2 (together with another binary alloy of titanium-germanium used for comparison in Table 1). (p.65, PDF p.15)
- 6.25 The titanium-germanium alloys of various compositions appear important when comparing the results from these alloys to the NiTi system. In Table 16 (p.16), heat treatment of the alloy at different high temperatures ranging from 1450°C to 1700°C and quenched rapidly to lock in these crystalline structures and how they can potentially change at different temperatures and composition of germanium is shown. In this case, we see a transition from the alpha crystalline phase at low temperatures into the beta crystalline phase at high temperatures, with a combination of the two phases of various percentages indicated in Table 16 (p.66, PDF p.16) within a certain temperature range. The authors were also trying to see how the combination of these alpha and beta structures would vary with increases in the quantity of germanium added to titanium.¹⁴

¹³ Does this mean Battelle and/or USAF realised TiZr at this composition range is a shape memory alloy and didn't want anyone to pursue the alloy for further study? Could this be the reason for monitoring the work of NYU scientists on TiZr in 1965?

¹⁴ It is clear at this point that the presence of two crystalline phases in a single piece of a titanium-base alloy was of interest to Battelle scientists and ultimately to the USAF by October 1949. Yet if this work had been carried out before early July 1947 when we assume the military had been testing a titanium-base shape memory alloy of some type, all this would not be news to the scientists or the military. Yet it is considered new information by October 1949 through the Second Progress Report. This could have important implications for the Roswell case.



- 6.26 A tentative phase diagram for the titanium-germanium alloy system is revealed on p.67, PDF p.17.
- 6.27 The same heat treatment and rapid quenching was applied to the nickel-titanium alloy when showing the extent of the combined alpha and beta crystalline phase field. We see this in Table 17 (p.69, PDF p.19)¹⁵. A third gamma phase is also included due to the fact that a nickel-rich phase is formed as clusters within the titanium beta phase matrix as the amount of nickel added to titanium exceeds above 7.3 per cent. A tentative phase diagram for nickel-titanium showing the different phase fields is revealed in Figure 20 (p.70, PDF p.20).
- 6.28 The authors stated the addition of nickel “markedly limits the alpha field and lowers the beta solvus line” (p.68, PDF p.18) and that it is probable for a eutectoid to exist between 6 and 7 per cent nickel.
- 6.29 There is no indication in the report to suggest further study into NiTi would take place in the future. According to the authors, they concluded the study by stating: “...the data do not justify further investigation of binary titanium-germanium or titanium-nickel alloys” (p.68, PDF p.18).¹⁶
- 6.30 The same combined alpha and beta crystalline structures locked in through rapid quenching was observed in the titanium-silver (NiAg) alloys between 1550°F and 1650°C (see Table 20 on p.76, PDF p.28). Initially the work into this alloy system involved 1 to 2 per cent silver according to the Summary Report, Part III (p.68, PDF p.18). It was extended for this report in the range 2.5 to 5.0 per cent silver. This system displays more similar crystalline structure phase results to NiTi including not just simultaneous presence of alpha and beta structures but also a gamma phase of silver-rich regions within the titanium matrix as seen in Table 17 (p.69, PDF p.19) together with a tentative phase diagram in Figure 20 (p.70, PDF p.20).

¹⁵ Using percentages of the alpha and beta phases in the same way for the nickel-titanium alloy as for the germanium-titanium alloy would suggest the USAF and Battelle were trying to make comparisons between the two alloys as well as show the extent of the dual crystalline phase.

¹⁶ Is this to discourage other scientists from looking too closely at these alloys?



- 6.31 The authors noted one difference between NiTi and NiAg. They wrote “It is evident the silver raises the beta and lowers the alpha solvus lines”. (p.74, PDF p.26). This observation is opposite in NiTi.
- 6.32 Again we find the same simultaneous alpha and beta phases being present in various percentages when composition exceeded 5 per cent zirconium and when the TiZr alloy is rapidly quenched from a temperature between 1450°C and 1650°C as revealed in Table 21 (p.83, PDF p.35). A tentative phase diagram for the titanium-zirconium system is shown in Figure 29 (p.84, PDF p.36).
- 6.33 There is effort to observe the mechanical properties of various titanium-base binary and more complex ternary alloys except for the nickel-titanium system. The reason why NiTi is not included in this work is because the mechanical properties were studied and the results mentioned in the previous bimonthly report.
- 6.34 The titanium-silver (pp.68-77 or PDF pp.18-29), titanium-beryllium (pp.78-80 or PDF pp.30-32) and titanium-zirconium (pp.80-84 or PDF pp.32-36) alloys were measured for their tensile strength, hardness and ductility.
- 6.35 Another interesting aspect of this work involved the aging (or hot-rolling over a period of time) for 4 hours in order to test the hardness. The authors learned the titanium-silver alloy “did not increase the hardness” (results revealed in Figure 25) when hot-rolled for 4 hours at 750°C p.26. Interestingly the same technique was not applied to the nickel-titanium alloy for comparison purposes.¹⁷
- 6.36 In the case of titanium-zirconium, the authors “concluded that titanium-zirconium alloys in the range of composition investigated do not show any significant response to heat treatment or aging”. In other words, these alloys would not show significant hardening with heat treatment and aging.¹⁸

¹⁷

It is well-known for NiTi to significantly increase its hardness when cold-working the alloy.

¹⁸

In the context of the dark-greyish titanium-based shape memory Roswell metallic foil, it is claimed by a military witness that shooting at the piece of this foil with a gun would not produce a hole suggesting the hardness was probably very high (as well as being extremely lightweight). Perhaps it is possible to eliminate titanium-zirconium as a possible candidate for the foil as heat treatment and aging of the alloy



- 6.37 Similar mechanical tests were performed when adding small amounts of columbium or tantalum in the range 1 to 2 per cent with titanium (p.85, PDF p.37)) — the elements would “increase the tensile strength and lower the ductility” of titanium. (p.61, PDF p.11).
- 6.38 Increasing the columbium content in titanium does increase hardness but no significant improvements to the hardness was observed when hot rolled and aged for 4 hours at 750°C. (p.85, PDF p.37).
- 6.39 A similar increase in hardness observed when combining around 1 to 2 per cent tantalum with titanium. It is claimed a future report looking at the titanium-columbium and titanium-tantalum alloys at higher composition range of the alloy additions (up to 10 per cent) would be considered. (p.88, PDF p.40)
- 6.40 The study of tensile strength properties expanded to include ternary alloys of manganese and carbon, manganese and vanadium, and molybdenum and tungsten (p.88, PDF p.40). It is here that the authors realised the alloying of metals using the arc melting technique was not properly combining the elements with titanium in order to provide homogeneity in the distribution of the elements throughout titanium (eg. manganese). And applying higher temperatures would not help the situation. The authors emphasise the importance of a uniform distribution of elements such as manganese throughout titanium when getting consistent tensile strength and elongation results from the same alloy sheet (p.88, PDF p.40). They also note the importance of “intermediate alloy hardeners for the introduction of the alloy additions rather than the use of pure metals during arc melting appears very promising and will be investigated more fully” (p.92, PDF p.44). In other words, a new melting technique of combining selected pure elements with pure titanium had to be employed and would involve the use of “master alloys, or hardeners, for introducing the alloying additions”. The authors claim “This

does not increase its hardness. Yet the nickel-titanium alloy when cold-worked and aging is applied does increase hardness significantly.



[new] melting technique will be used more extensively in the future” (p.61, PDF p.11).

- 6.41 Point 6.23 would suggest the proper alloying of elements to titanium was still in its infancy stage for the USAF and Battelle by 1 September 1949 and only realised a more effective technique to combining elements with titanium by 31 October 1949. The work of these authors on finding at least one high-strength titanium-base alloy indicate: “Steps have been taken to re-evaluate the more promising high-strength alloys. This work is directed toward selecting an alloy composition on which extensive engineering data will be obtained” (p.61, PDF p.11).
- 6.42 Alloys of greatest interest following this study are those principally containing high tensile strengths and hardness. At the time the study was made, the best alternative non-NiTi alloys are those containing vanadium. However, remarkably it does not mention the high hardness and tensile strengths to be found in NiTi when cold-working the alloy. It suggests the USAF and Battelle were looking for alternative titanium-base alloys with favourable mechanical properties as those of NiTi without mentioning the results of mechanical tests for NiTi.



7

Section titled Investigation of Refractories for Melting Titanium, by P.D. Maddex and L.W. Eastwood

7.8 The authors were looking for new materials to make high temperature crucibles for holding molten titanium and its alloys. The report stated:

“Tests were completed on the evaluation of “hot-pressed” titanium carbide and graphite crucibles lined with tantalum carbide and tungsten boride as refractories for molten titanium. Melts were prepared in crucibles made of zirconium oxide (stabilised with CaO), calcium oxide, calcium oxide fluxed with TiO₂, and aluminium oxide. The stabilised zirconium oxide crucible was the first refractory tested which had areas not wet by the molten titanium. Therefore, additional melts in this type of crucible are planned to evaluate this refractory further. None of the other refractory materials tested appear to be useful.” (p.61, PDF p.11)¹⁹

7.9 The process for producing pure titanium involved “standard specimens” of the iodide titanium form. These specimens had to be tested during the period of this report to see how susceptible they were to absorbing oxygen impurities by adding “known amounts of oxygen...as TiO₂” (p.62, PDF p.12). This aspect

¹⁹ This quote suggests by October 1949 that USAF and Battelle were still using crucibles to melt limited quantities of pure titanium combined with other elements in a vacuum furnace. It is therefore highly unlikely the USAF or Battelle had the manufacturing capacity or technology to produce adequate quantities of the shape memory metallic foil observed by firsthand Roswell witnesses at any time before July 1947. It means the USAF and Battelle could not be the original inventors of the metallic foil and of the object that crashed near Roswell. This would imply the object is not owned by the US military or any scientific establishment on American soil. And since the titanium industry began in the US by USAF after 1947 and with no other country coming forth to claim responsibility for the titanium-based shape memory alloy object, then it would appear we may have evidence of an exotic artificial object picked up by the US military.



of the work and results obtained was “prepared and submitted to Dr G. Derge of the Carnegie Institute of Technology for vacuum-fusion analysis” (p.62, PDF p.12).

7.10 These crucibles were not considered very large. Large quantities of pure titanium and titanium-base alloys were not established even by 31 October 1949.²⁰

²⁰

This raises serious questions as to how the USAF and Battelle had managed to produce large quantities of a dark-greyish titanium-base shape memory alloy near Roswell in early July 1947.



8

Section titled Analytical Methods for Titanium-base Alloys: Studies on the Chemical Analysis of Oxygen in Titanium by the Chlorine-Carbon Tetrachloride Method, by E.J. Center and A.C. Eckert

- 8.13 The previous bimonthly report gave “a brief discussion of the chlorine-carbon tetrachloride method for determining oxygen” (p.97, PDF p.55). This report explains “the apparatus and techniques used in the investigation of this proposed analytical method...”
- 8.14 A discussion of the future work by these authors suggests there is interest in high-strength titanium alloys and to choose the best one for producing in large quantities of ingots exceeding 2 pounds (p.119, PDF p.77).²¹
- 8.15 The report claims more work on test crucibles and investigation of refractory materials will continue (p.119, PDF p.77).

²¹ The aging and cold working of NiTi would constitute an excellent high-strength titanium alloy due to the way it naturally hardens. Yet remarkably this was not explored in this or any other report as far as we can tell. Furthermore, the choice of a high-strength alloy of reasonable hardness would only be produced in large quantities (ie. “...relatively large ingots” exceeding 2 pounds) once 2-pound ingots of “several selected alloys” were made and evaluated for their “fabrication characteristics” as well as strength. This further supports the view that large quantities of a titanium-based shape memory alloy was not being produced before July 1947.



Summary

NiTi or nitinol is of tremendous importance to the USAF at Wright-Patterson AFB in the First Progress Report and in the last report for contract number W 33-038 ac-21229. It would continue into the Second Progress Report.

Looking at the formerly classified USAF/Battelle First and Second Progress Reports in the second stage development of titanium-based alloys, we see no use of the term *martensite transformations*, *pseudoelastic alloys*, or other similar technical terms. Even the composition range chosen for NiTi suggests they were not interested in the equi-atomic region where the shape-memory effect is most noticeable at room temperature. This may lead us to suspect the USAF and Battelle were probably not aware of shape-memory alloys at the time. Yet no amount of careful positioning of the final composition range for this study—in this case at the titanium-rich end of the composition range for NiTi—or lack of specific terms to suggest a shape-memory effect can deny the possibility they did know. The work of Craighead, Eastwood, and Fawn in the report clearly emphasizes the establishment of a dual crystalline phase region for various alloys, including nitinol. This is crucial information. The existence of a dual crystalline phase is paramount to determining a potential shape-memory effect in any alloy.

When combined with witness claims of a shape-memory nitinol-like Roswell foil that ended up at Wright-Patterson AFB for analysis, it becomes more than just a possibility...it is highly probable the USAF and Battelle did know.

If this does not seem sufficiently compelling, the reports show Battelle had been requested, under contract with USAF, to perform alloy research on no less than two known shape-memory alloys—NiTi and TiZr. Moreover another shape-memory alloy—NiTiCo—is believed to have been mentioned but was removed from this



report (i.e., missing pages), with the potential for NiTiCr to be another. In all, we have no less than three, and potentially up to four, shape-memory alloys in the one report—specifically the Second Progress Report of October 31, 1949. And it isn't the only report to reveal a study into NiTi and other shape-memory alloys.

For what is supposed to be an ordinary alloy, NiTi was given more than just a cursory examination. The First Progress Report for contract AF 33(038)-3736 claims the work into the alloy went back to the previous contract number W 33-038 ac-21229 (the first contract on titanium to be issued by the USAF with Battelle after 1947), as if suggesting the alloy was the principal reason for issuing the contract with Battelle. And all the while, in both the First and Second Progress Reports, the USAF and Battelle tried to claim that no further study of NiTi was warranted or required as if to encourage other scientists not to learn more about NiTi, but they still studied it for some reason.

It makes one wonder how many more titanium-based shape-memory alloys, including NiTi, we going to find if all Battelle/USAF Progress Reports for the period from 1948 to 1949 are released for public scrutiny for both USAF/Battelle contracts. If all this work was meant to be a coincidence, it is a remarkable one.

The First and Second Progress Reports may not prove or disprove conclusively the USAF had direct knowledge of shape-memory alloys. Nor does it prove NiTi is or is not the alloy the witnesses observed in the Roswell foil. However, on the basis of probabilities, and the fact that too many witnesses reported a shape-memory effect in the nitinol-like Roswell-foil that ended up for analysis at Wright–Patterson AFB, it is highly unlikely the USAF were not aware of shape-memory alloys at the time.

In addition, it would be astonishing if NiTi were not somehow linked in some way to the Roswell foil, given the considerable attention given to it by the USAF and Battelle at the time. Furthermore, the dark-grey color of the Roswell foil is virtually the same as NiTi. The fact that we cannot identify another dark-grey shape-memory alloy containing titanium can only raise the prospect that NiTi or a similar alloy containing significant amounts of nickel and titanium could well be the foil the witnesses observed.

It is highly recommended that all previous reports into titanium development and research be declassified and analysed to show exactly the extent of interest in NiTi



and other shape memory alloys, as well as the reason why titanium alloys became of great interest to the USAF after 1947.

And, certainly it is imperative at this stage of the Roswell research to obtain the first progress report of contract number W 33-038 ac-21229 as this appears to be the earliest known report when the work into titanium research ever began on an official classified level. The date of the report is likely to be 30 June 1948 covering the period from 18 May to June 1948.