# Detecting and Characterizing Overtemperature Exposure in Titanium Via Process Compensated Resonance Testing

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#### ABSTRACT

This paper presents the results of an evaluation of Process Compensated Resonance Testing (PCRT), as well as other Non-Destructive Inspection (NDI) evaluations, for the detection of heat damage in titanium material used for aircraft brake torque tubes. This study reviewed PCRT, ultrasonic testing (UT), eddy current testing (ET), hardness, and color evaluation of Titanium 6AL-4V (Ti-64) samples before and after various heat treatments. Each NDI method was compared by examining the relationships between the measurements made by each of the methods to both the temperature parts were exposed to and the results of mechanical testing on the coupons. PCRT was highly sensitive to Ti-64 thermal history and, of the NDI methods examined, had the best correlation to the coupon mechanical performance. Two PCRT Pass/Fail inspections were configured and demonstrated detection of heat damage in less than 4 seconds per part.

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#### INTRODUCTION

Torque tubes are a critical component of aircraft braking systems. Designed to absorb the torsional and thermal loads produced by the brake system, a torque tube is a heavily stressed component whose material properties must meet the required specification. Titanium's mechanical and thermal properties make it an excellent material for aircraft brake torque tubes, but it is not impervious to heat damage from extreme braking events. Degradation of material properties and in-service failure may result if heat-damaged torque tubes are not detected and removed from service. Some references suggest titanium alloys for aerospace applications have good mechanical performance at operating temperatures up to 600° C. Above that temperature most titanium alloys will rapidly soften, creep, and/or oxidize [1]. Often the titanium alloys will change color after heat exposure due to varying properties of the titanium oxide layers on the surface of the part. For instance, when Ti-6AL-4V (Ti-64) components are exposed to temperatures between 90 - 480° C, white TiO and gold TiO<sub>2</sub> oxides form on the part's surface. Temperatures between 480 - 680° C favor the formation of blue Ti<sub>2</sub>O<sub>3</sub> and green Ti<sub>3</sub>O<sub>3</sub> oxides and temperatures < 680° C cause an inward diffusion of oxygen that produces a hard, brittle, and porous surface layer that appears dark grey [1]. The observed color change of the material is influenced by the temperature, distribution of heat around the object, the amount of time held at a specific temperature, and the thickness of any previous oxide layers due to its thermal history. While a visual color evaluation is often employed as a Non-Destructive Inspection (NDI) for the detection of heat damage in titanium components, it is subjective, qualitative, obscured by part

cleanliness and surface residue, and has not been found to have a simple linear relationship between color and thermal history. Titanium torque tubes often build up carbon dust from brake pads during their time in-service, further obscuring the surface color evaluation. An objective, quantitative NDI inspection that reliably and accurately detects thermal damage in the midst of confounding factors is needed.

Heat treatment and quenching of Ti-64 between 600 -1200° C can produce as much as 10% variation in Young's Modulus (E) due to composition and changes in the volume fraction of the constituent material phases [2]. Changes in E will make the material stiffer or softer and thus increase or decrease the part's natural resonance frequencies. Process Compensated Resonance Testing (PCRT) uses the collection and analysis of a part's resonance frequencies as a basis for inspection[3][4][5]. PCRT has proven sensitive to the material changes caused by heat damage, earning FAA approval for inspections for heat damage in gas turbine engine blades [6]. PCRT inspection also requires no part cleaning, as a part's resonance frequency is unaffected by paints and surface dirt.

In the present work, the ability of PCRT to detect heat damage in titanium coupons, compared to other NDI techniques was investigated. NDI methods evaluated in this study included PCRT, Eddy Current (ET), Ultrasonic (UT), Vickers hardness testing, and color evaluation. Destructive evaluation of coupon material properties occurred through tensile testing and the relative effectiveness of each NDI method was assessed by examining the relationships between the inspection results, thermal history, and mechanical testing results.

#### MATERIALS AND METHODS

Rolled Titanium Ti-64 bar stock, that had a heat treatment of 700° C for 2 hours followed by an air quench, was machined into 54 ASTM E-8 [7] compliant dogbone coupons (0.64 cm diameter x 7.62 cm length). These Ti-64 coupons were heat treated from 100 - 800° C with the intention of causing both acceptable and unacceptable microstructure changes. Coupons were placed in a room temperature kiln, heated to the target temperature, left to soak at temperature for 30 min, and then removed from the kiln to allow for air quenching. A selected number of samples were also heat treated with a more complex thermal history (including longer soak times, progressively increasing and decreasing temperature treatments) to be evaluated at a later date. All coupons were evaluated before and after heat treatment with each NDI method. Samples that did not receive any heat treatment were evaluated twice to estimate measurement error of each NDI method.

A PCRT laboratory-grade test fixture was constructed to measure the resonance spectra of each coupon (Fig. 1). The coupons were placed in a v-block of four leadzirconate-titanate piezoelectric (PZT) transducers and a guide screw was used to repeatedly place the coupons in the same horizontal position. One transducer, the 'drive', excited the part with a swept sine waveform. Two transducers, the 'receive' channels (Ch1 and Ch2), recorded the resonance response of the part. A fourth transducer was used to balance the coupon in a stable position. The resonance spectrum between 10 kHz and 280 kHz was recorded, after being compensated by the part temperature, and 30 resonance peaks were identified for analysis.

Any color change on the gauge section of the coupons after heat treatment was evaluated based on a visual inspection. Spectrophotometry methods [8] were not employed in this study, however, they can allow for a more precise analysis of the macroscopic color on the surface (by measuring the reflectance and/or transmittance spectra of the samples). A Vickers hardness tester was used to measure the hardness on both flat faces of the E-8 coupons, in compliance with ASTM E92-17 [9]. The tester used a 10 kg load with a dwell time of 15 seconds and made three indents on each face. The indent depths were measured optically, and the values converted to a Vickers Hardness Number (VHN). The average VHN hardness value for each face was recorded.

Using an ET Olympus Nortec 600 conductivity meter and Nortec SPO-887 60 kHz probe the electrical conductivity of each coupon was measured. ET readings involved a direct measurement on the gauge section and the flat end of the dogbone which displayed the conductivity of the metal in %IACS (% International Annealed Copper Standard). An Olympus Epoch XT detector with a 0.64 cm/ 5 MHz transcoder probe was used to record UT longitudinal velocity measurements at three spots along the long axis of each coupon.

Tensile testing was included in these studies to provide more direct information about the changes in material property that occur in Ti-64 due to heat exposure. A selected number of heat treated and non-heat treated samples were sent to an outside vendor for tensile testing in compliance with ASTM E-8 [7] using a 100 kN Instron tensile screw driven rig. Results included a reporting of each part's ultimate tensile strength (UTS), yield strength (YS), Tensile Modulus (TM), and percent area reduction (RA).

#### **RESULTS AND DISCUSSION**

#### Tensile Testing

The tensile results showed a high sensitivity to thermal history in titanium. Fig. 2 presents the measured UTS, YS, TM, and RA vs heat treatment temperature for several non-heat treated and 30 heat treated coupons that had single heat treatment exposure. The dashed black lines were the nominal variation of results found from the tensile testing of 7 non-heat treated coupons. Parts heat treated up to 400° C showed similar results to the non-heat treated parts. Parts heat treated between 450° - 550° C showed an increase in strength while parts heat treated between 600° - 800 °C showed a decrease in strength (UTS and YS) and TM. However, parts treated at 650° C showed the widest range of tensile results across all measurements.

#### **Color Evaluation**

Before heat treatment, all coupons had a shiny grey surface and no color change was observed after heat exposures from 100 - 300° C. Heating parts from 300° - 800° C showed color changes progressively from silver to yellow straw, blue, purple, dark grey, bronze, and copper. Table 1 summarizes the color change vs. heat treatment temperature for parts that had a single heat treatment with a 30 min soak time. While the gauge section of the coupon was often a uniform color, a few parts showed multiple colorings on the thread sections and the flat end faces compared to the color change on the gauge section. Variations in heat treatments performed around 650° C caused the

widest spread in the tensile testing results, yet all these parts turned the same dark grey color. In field inspection, the dark grey would be harder to distinguish from a part exposed to lower temperatures, because of normal dirt, oils, and in-service use. Even though the part changed from silver, light straw, to dark straw color between 200-500° C, no YS or UTS changes were seen in this range. However, the blue surface color produced above 500° C did correlate to an increase in YS and UTS. Also, the copper color produced after exposure to 800° C did correlate with the parts with the lowest UTS.

#### Hardness Evaluation

Only a few parts subjected to heat treatments showed changes in average VHN hardness beyond measurement error. Fig. 3 presents the relationship between % change in average VHN and both the heat treatment temperature and resulting YS values. The measurement error was calculated experimentally from multiple measurements of parts that did not receive any heat treatment, and is represented by the dashed black lines in Fig. 3 . Only one part heat treated to 750° C showed a decrease in hardness of over 15% in VHN while other coupons heat treated to the same temperature did not show the same hardness decrease. Due to the measurement error for the hardness results, a lower correlation of hardness to tensile properties (R<sup>2</sup> value of 0.15) was observed in the temperature ranges examined. Similar results showing poor correlation of hardness and tensile data have been observed for a variety of other titanium alloys **Error! Reference source not found.**. Hardness changes can occur after heat treatments in titanium because of microstructural alpha-beta precipitation changes

at different heat exposures [10] yet, the poor correlation here was attributed to the difference in prevailing deformation mechanisms in hard testing (microtwining) compared to the tensile testing (shear) **Error! Reference source not found.**.

#### UT Evaluation

The velocity of ultrasonic waves can be used to assess the microstructure state of material as the wave speed can be affected by the material's phase composition, grain size, and phase anisotropy which can change after part heat treatment [11]. Changes in the UT wave propagation speed are often used to correlate to a material's E [11]. Fig. 4 shows the average % change in velocity of parts treated at one temperature compared to their heat treatment temperature. Measurable change was seen outside measurement error (dashed line) for some parts exposed to > 500° C, and for several heat treated below 200° C. Other parts, including some heated beyond 650° C, showed almost no change. However, the error bars in the UT velocity measurements are on the order of  $\pm 0.5\%$ . In this study, the higher measurement error in the UT results produced a relatively low correlation (R<sup>2</sup> value of 0.05) of UT velocity to tensile performance. Fig. 4 shows the correlation of the measured YS and the % change in average measured UT velocity before and after heat treatment. Comparison of UT velocity to UTS and modulus showed similar results.

#### ET Evaluation

The ET results on the samples were inconclusive. The curved surface on the coupon gauge section put limitations on where conductivity measurements could be made, so conductivity measurements were made on the flat ends of the dogbone. Yet, the flat surfaces had a small hole in them, which created an air gap that influenced the conductivity results. The same reading of 1.0% IACS was obtained for all coupons in both the before and after heat treated states and the % change in conductivity was zero for all samples, regardless of heat treatment temperature. If future ET measurements are required on these dogbones, a coupon test fixture should be created to ensure that the eddy current probe is placed in the same position on each piece and a curvature correction factor should be used. In this fixture, the probe would move slowly and gently across the curved gauge section at right angles (to the long axis) and the maximum value indicated could be corrected with the appropriate curvature factor. Eddy current measurements on a thicker Ti-64 geometry, like those on sections of a torque tube, should yield more conclusive results on an ET method viability for detecting heat damage.

#### PCRT Evaluation

The resonance frequency changes for all samples heated to the temperature intervals above 450° C were well beyond the PCRT measurement error range for nearly all the resonance modes, indicating high PCRT sensitivity to thermal history in titanium. Fig. 5 shows the % frequency change vs heat treatment temperature for three different resonance modes between 117 - 119 kHz (Peaks 15, 16 17) with the PCRT measurement

error represented by dashed black lines. Fig. 5 also shows the linear correlation of the measured YS and the % change in one PCRT frequency before and after heat treatment for all samples sent for tensile results. The resonances of parts heat treated up to 400° C showed no effective change while parts heat treated between 450° - 550° C showed an increase in frequency. Parts heat treated to between 600° C - 800 °C showed a decrease in resonance frequency.

The pattern of the tensile material property change of YS and UTS vs temperature was very similar to the pattern seen in the PCRT results vs temperature. Even though measurement error was present in the tensile and NDI measurements, PCRT results showed the highest correlation of tensile properties compared to the other quantitative NDI methods in this study (ET, UT, and hardness) with an R<sup>2</sup> value of 0.65. The trend of R<sup>2</sup> correlation values for PCRT and tensile results ranged from 0.58 - 0.65 depending on which peak was used. Similar correlation results were seen with the correlation of PCRT to UTS. As with the tensile results, parts heat treated at 650° C showed a range of PCRT values.

#### PCRT Inspection Demonstration

In a PCRT Part-to-Itself (PTI) inspection, the change in frequency was calculated for each part based on its before and after heat treatment resaonce frequencies. To configure a PCRT PTI inspection to detect allowable change due to heat exposure, limits were placed on the allowable frequency change for three resonance peaks. While control limits can be set based on several factors, including the heating threshold

correlating to excessive heat damage, an arbitrary limit of 650° C was chosen for this study, as manufactures specifications for heat damage in Ti-64 torque tubes was not available. If a lower temperature, such as 500° C, were found to be critical, the limits could be adjusted to reflect this. Fig. 6 shows the results of the PCRT PTI inspection with control limits set to fail parts exposed to temperatures greater than 650° C. If a part had measured frequency changes between 0 and 0.5% for the 3 diagnostic resonances in the inspection, corresponding to cumulative heat damage below the 650° C threshold, the part passed the PCRT inspection. If the part had a change greater than  $\pm$ 0.5%, the part failed inspection. Measured frequency changes that indicate heat damage at or above 650° C, with a decrease in frequency, produced a reject result. While this PCRT inspection requires parts to be measured in both a *before* state (new or in-service) and *after* state (service interval or repair state).

In cases where both a before and after state measurement is not possible, a Targeted Defect Detection approach will measure the frequencies in only the *after* state to determine if heat damage has occurred. A PCRT Targeted Defect Detection inspection was created by training a set of Vibrational Pattern Recognition (VIPR) algorithms to identify resonance peak patterns that would fail parts exposed to temperatures greater than an arbitrary limit of 650° C. The VIPR algorithms identified peak frequencies that distinguished between classified acceptable or unacceptable parts. After the peaks were identified, the peaks for each part were scored using a Mahalanobis-Taguchi system approach (MTS) which scores the parts relative frequencies to the central tendency of

the acceptable parts (in N dimensional space, where N is the number of peaks used). After placing the part on a PCRT fixture, this inspection took approximately four seconds to scan a part, automatically calculate an MTS score, and return a pass/fail result. Fig. 7 shows the MTS results vs. heat treatment temperature for all the coupons. The solid vertical dashed line was the set inspection limit of 650° C. This inspection passed all the non-heat treated parts, as well as parts that experienced lower temperature heat treatments between 100- 450° C. This inspection failed all parts that were heat treated >650° C. While most of the parts heat treated between 500°- 650° C passed inspection, several parts in this range failed. Some of these failures could be the result of inconsistences in the heat treatments such as fluctuations in air quenching temperature or equal circulation of air in kiln during treatment. The VIPR-based sorting was more sensitive to population variability than the PTI measurement approach, so some extra conservatism was built into the algorithm. This led it to failing some of the coupons heat treated to temperatures that approached the 650° C threshold. For VIPR-based inspection, the pass/fail limits and VIPR training criteria can be adjusted to optimize sensitivity and yield. The PTI measurement approach, because it is based on the frequency change in individual components, removes part-to-part differences as a variable, and requires less conservatism. The two PCRT inspections demonstrated the ability of PCRT to determine if the TI-64 part were in an acceptable material state. PCRT also has the potential to quantitatively approximate the temperature/heat exposures the parts have experienced by examining the absolute and change in resaonce frequencies of a part.

#### CONCLUSION

This study produced strong correlations between temperature exposure, ultimate tensile strength, and the PCRT resonance frequency shifts for Ti-64 coupons after various heat treatments ranging from 300 °- 850° C. Two PCRT Pass/Fail inspections were configured based on the absolute resonance frequency of a part as well as the change in frequency before and after heat treatment. Additional NDI methods of color, hardness, UT, and ET were also employed in this work to assess their capabilities in measuring temperature exposure. Color change after heat treatment was subjective, while hardness and UT evaluations had high measurement error and showed less correlation to tensile results than PCRT. ET results were inconclusive due to the geometry of the dogbone coupons used in this study. Future work will expand evaluations to include larger populations of sample coupons with more complex thermal damage scenarios such as localized heating and varying exposure time, with the addition of metallography evaluation to quantify the microstructure changes after heat treatment. Future studies will also apply PCRT to the evaluation of Titanium aircraft brake torque tubes with thermal damage.

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## NOMENCLATURE

ET	Eddy Current Testing	
MTS	Mahalanobis-Taguchi Score	
PCRT	Process Compensated Resonance Testing	
PTI	Part-to-itself Inspection	
Ti-64	Titanium Alloy Ti-6AL-4V	
ТМ	Tensile Modulus	
UT	Ultrasonic Testing	
UTS	Ultimate Tensile Strength	
VHN	Vickers Hardness Number	
VIPR	Vibrational Pattern Recognition	
YS	Yield Strength	

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# **Figure Captions List**

Fig. 1	PCRT fixture diagram
Fig. 2	Tensile results vs heat treatment temperature; (a) 0.2% YS; (b) UTS; (c)
	TM; (d) RA
Fig. 3	Hardness results; (a) % change in VHN vs heat treatment temperature
	and (b) % change in 0.2% YS vs % change in VHN
Fig. 4	UT results; (a) % change in average UT velocity vs heat treatment
	temperature and (b) 0.2% YS vs % change in UT velocity
Fig. 5	PCRT results; (a) % change in 3 resonance modes vs heat treatment
	temperature and (b) 0.2% YS vs % change in a single resonance mode
Fig. 6	PCRT PTI inspection results using % change of frequency measurements
	before and after heat exposure
Fig. 7	PCRT VIPR MTS inspection results vs heat treatment temperature

# **Table Caption List**

Table 1Color change after heat treatment



Fig. 1 PCRT fixture diagram



Fig. 2 Tensile results vs heat treatment temperature; (a) % 0.2 YS; (b) UTS; (c) TM; (d)



Heat Treatment Temperature, °C	Coupon Color
As received	Silver
100	Silver/No Change
200	Silver/No Change
300	Silver/No Change
400	Light Straw
450	Dark Straw
500	Dark Straw
550	Blue
600	Purple
650	Dull Grey
700	Bronze
750	Bronze
800	Copper

Table 1 Color change after heat treatment



Fig. 3 Hardness results; (a) % change in VHN vs heat treatment temperature and (b) %

change in 0.2% YS vs % change in VHN



Fig. 4 UT results; (a) % change in average UT velocity vs heat treatment temperature and

(b) 0.2% YS vs % change in UT velocity



Fig. 5 PCRT results; (a) % change in 3 resonance modes vs heat treatment temperature

and (b) 0.2% YS vs % change in a single resonance mode



Fig. 6 PCRT PTI inspection results using % change of frequency measurements before

and after heat exposure



Fig. 7 PCRT MTS inspection results vs heat treatment temperature