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Dimensional stability of cryotreated diaphragms of pressure transducers

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Abstract. Accurate data of propellant pressures are very critical for the successful propulsion of launch vehicles. These pressures are measured using the integral diaphragm pressure transducers machined from precipitation hardened martensite stainless steel (APX4). The thin diaphragms of these transducers gradually develop dimensional changes with time due to the conversion of residual austenite to martensite, resulting in drift in output pressures. This situation calls for enhancement of dimensional stability of the diaphragms which is carried out by cryotreating them at 98K for 36 hours followed by tempering at 673K for one hour in vacuum furnace. The dimensional studies were carried out on three categories of diaphragm materials viz. normal, cryotreated and cryotreated followed by tempering using the Thermo Mechanical Analyser (TMA). The dimensional changes of the test specimens were determined at intervals of 5K in the temperature range from 123K to room temperature. The results are analysed and presented in this paper.

1. Introduction

The Measurement of propellant pressures is very critical in the propulsion of launch vehicles. These measurements are carried out by using pressure transducers. Among the various types of pressure transducers, the integral diaphragm pressure transducers are widely used in space applications [1]. These transducers are produced by machining precipitation hardened martensite stainless steel (APX4). In this transducer, the transducing element is an integral metallic diaphragm with bonded foil strain gauge. When the propellant pressure is applied to one side of the diaphragm, the resulting strain is sensed by the strain gauge which is mounted on the other side. This strain is converted into measurable electrical outputs mounted through Wheatstone bridge networks which are calibrated in pressure units. These transducers are expected to exhibit dimensional stability over the entire useful life for reliable performance and dependability.

The precipitated hardened martensitic steel used for machining of pressure transducer does not contain complete martensite and a certain amount of unstable austenite known as retained austenite is generally present. This retained austenite is in metastable state and gets transformed to martensite when it is left alone which takes long duration spreading in months for the complete transformation. Martensitic transformations are first-order transformations and proceed through the localized nucleation and growth of individual crystals of martensite within austenite structure. Martensite plates typically form on multiple variants of the habit plane in a given grain of austenite. The freshly converted martensite has higher volume as compared with the original martensite and causes dimensional changes after long duration [2]. The diaphragm thickness values are very small and hence dimensional changes cannot be neglected. This causes zero shift and drift in the output readings. Experimental studies carried out with Vibrating Sample Magnetometry (VSM) techniques by Matteo et al. [3] have indicated significant reduction in time for the conversion to martensite structure when the steel specimens were subjected to cryogenic treatment. To enhance conversion of the austenite

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phase to martensitic structure, it was planned to cryotreat the diaphragms to study the effects on their dimensional stability. In the present study, the machined pressure transducers are subjected to cryogenic treatment at 98K for 36 hours using liquid nitrogen in a specially designed and developed cryotreatment unit. After the cryotreatment process, the diaphragms are tempered in vacuum furnace for one hour at 673K. The dimensional stability analysis of these diaphragms is determined with the help of a Thermo Mechanical Analyser over the temperature range from 123K to ambient.

2. Pressure transducer

The role of pressure transducer sensor used in space application is considered important from the point of sensing the propellant pressure. It incorporates an integral diaphragm made from precipitation hardened martensite stainless steel (APX4) to sense the input pressure. The pressure induced on one side of the integral diaphragm is converted into strain which is sensed by the bonded foil strain gauge mounted on the other side. This induced strain is converted into a measurable electrical output through a Wheatstone bridge circuit. The schematic diagram of the integral diaphragm pressure transducer is shown in Figure 1.

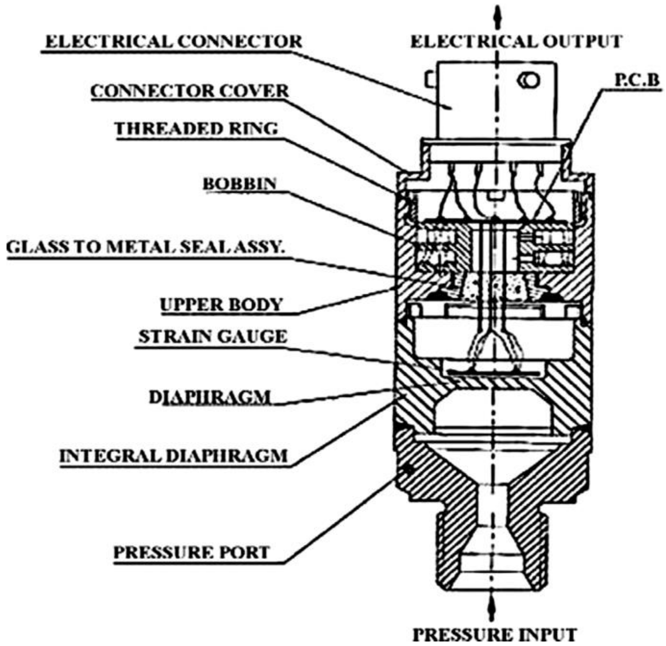

Figure 1. Schematic of integral diaphragm pressure transducer.

3. Cryogenic treatment

3.1. Cryotreatment process

The process of cryogenic treatment (normally known as cryotreatment) on metals has been used over many decades mainly in tooling industries to improve the life of cutting tools. It also finds application in many engineering activities like reduction of internal stress in metals, enhancement of dimensional stability of machined parts, improvement of the mechanical properties and thermal conductivity of selected metals, etc. [4]. A conventional cryotreatment cycle essentially includes three stages where the metal samples are cooled gradually to cryogenic temperature in a cryotreatment system, held for extended time duration and gradually warmed to room temperature. In a typical practice a total cryotreatment cycle takes around 50 hours with 5 hours for gradual cooling, 36 hours for soaking and 9 hours for warming up to room temperature. To achieve the cryogenic temperature liquid nitrogen is used as a cooling medium. A typical cryotreatment cycle is shown in Figure 2. The operating parameters of temperature and time can be varied to obtain optimum results. Normally, the cryotreatment cycle is followed by tempering for selected temperature and time.

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Integral diaphragm pressure transducer used for pressure measurement is manufactured from precipitation hardened martensitic steel (APX4). However, this steel contains a certain amount of retained austenite, which is a softer and more ductile phase of steel compared to martensite. Over time, the retained austenite slowly converts to martensite, resulting in dimensional changes due to differences in the coefficients of volumetric expansion [5, 6]. These dimensional changes are significant for the diaphragm, as its thickness is very small. Therefore, it is crucial to enhance the dimensional stability of the material to ensure reliable performance of the diaphragm in pressure measurement applications.

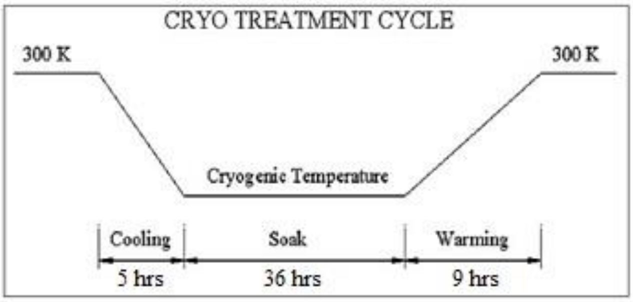

Figure 2. Cryo-treatment cycle

3.2. Cryotreatment system

The cryotreatment system incorporates mainly the cryotreatment chamber and auxiliary liquid nitrogen (LN2) supply manifold. Controlled quantity of LN2 is supplied to the chamber to maintain the desired temperature. LN2 supply to the chamber is regulated by using a solenoid valve which is activated by a PID controller. The system is designed and developed to produce cryogenic treatment to diaphragms by indirect cooling with no contact with LN2. This ensures no thermal shocks and cracks of the tools due to cold spots. The PID controller can be programmed for various cycles of cooling, soaking and warming. The temperatures of the specimens are measured using temperature sensors (PT100). The temperature data of the specimens are read by the PID controller continuously through the cryotreatment process. The schematic diagram of the cryotreatment system is shown in the Figure 3.

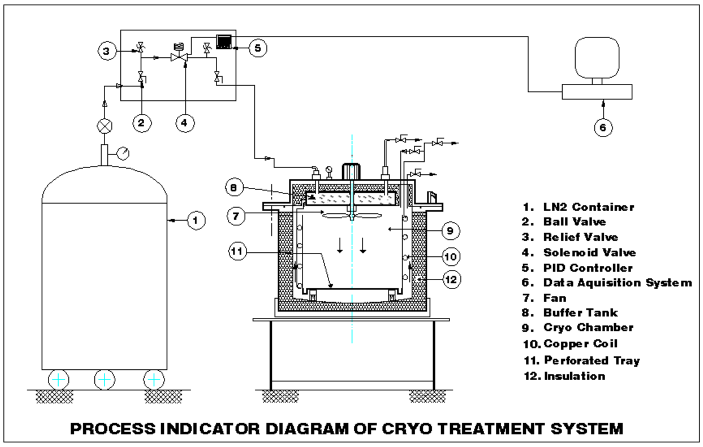

Figure 3. Process indicator diagram of the cryotreatment system

The cryotreatment unit is a double walled stainless steel container with the interspace filled with polyurethane foam. The top cover made of stainless steel has a double end shaft fan-motor assembly

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mounted centrally. The liquid nitrogen connecting valves, pressure gauges, feed through and outlet connections are all mounted on the top cover. Below the top cover, a cylindrical buffer tank is mounted where the supplied liquid nitrogen gets collected and evaporated vapours are vented through a vent pipe. The shaft of the fan motor assembly passes through this buffer tank and has fan blades mounted on both the sides. To ensure better heat transfer, a copper disc has been fixed to the bottom of the buffer tank. Aluminium fins are fixed to the bottom of the copper disc circumferentially to ensure forced convection cooling of the space inside the cryotreatment chamber. Figure 4 displays a photograph of the developed cryotreatment unit.

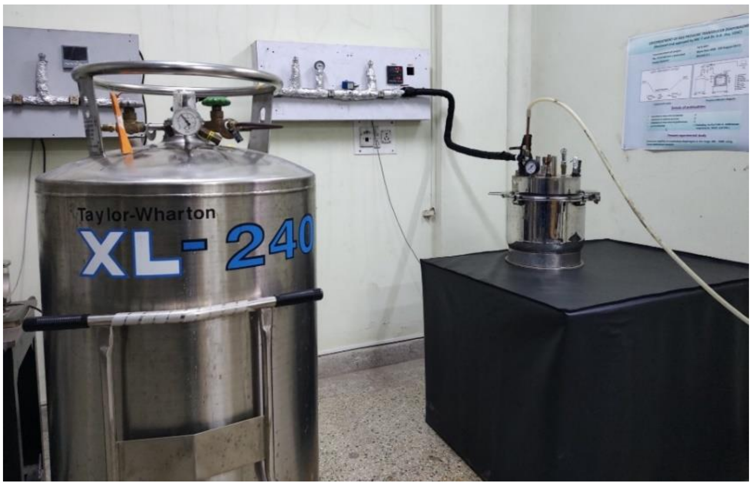

Figure 4. Photograph of the cryotreatment unit.

4. Experiments

The dimensional stability of the diaphragm material (APX4) is determined by analysing the coefficients of linear expansion using Thermo Mechanical Analyser. The values of coefficient of linear expansion were determined from cryogenic temperature (123K) to room temperature.

4.1. Thermo mechanical analyser

The objective of the experimental analysis is to determine the coefficient of linear expansion which is carried out using the Thermo Mechanical Analyser. Specimens in the form of a cube of sides 10 mm were machined using high accuracy milling machine ensuring parallelism between the end faces. These specimens were divided into three sets. Keeping the specimens of the first set untreated, specimens of the second set were cryotreated. Specimens of the third set were cryotreated at 98K for 36 hours and tempered in vacuum furnace at 673K for one hour.

The specimen is rigidly held inside the test chamber arresting its sideward movements. The chamber is cooled to cryogenic temperature with the help of liquid nitrogen. When the specimen reaches temperature of 77K, it is gradually heated with an electrical heater. The spring loaded plunger of a Linear Variable Differential Transducer (LVDT) tightly presses against the flat surface of the specimen and moves with it. The incremental changes in dimensions of the specimen are recorded with the help of DAQ system. The schematic of the system is shown in Figure 5 and the experimental setup of Thermo Mechanical Analyser is shown in Figure 6.

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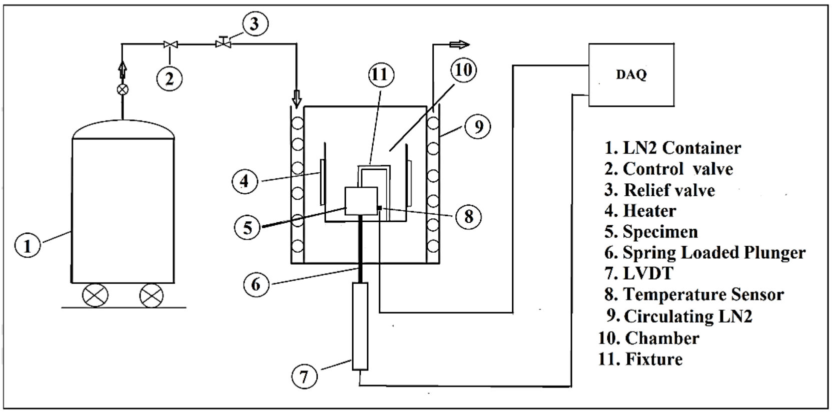

Figure 5. Schematic of the experimental system

Figure 6. Experimental setup of Thermo Mechanical Analyser

4.2. Procedure
After positioning the specimen, it is slowly cooled from room temperature down to 123K at a cooling ramp rate between 2 to 10K per minute using circulating liquid nitrogen. After reaching the steady temperature of 123K, the specimen is gradually heated to room temperature at a constant rate of 5K per minute with the help of an electrical heater which provides uniform and controlled heating. As the temperature increases, the length of the specimen increases proportionally and the readings of incremental lengths are recorded continuously at an interval of 5K. The inbuilt software of the system computes the values of coefficient of linear expansion and these values are continuously recorded and plotted by the Data Acquisition System (DAQ).

5. Results and discussion
The dimensional stability analysis is carried out by analysing three sets of samples as mentioned earlier. The values of coefficients of linear expansion were experimentally determined over the temperature range from 123K to room temperature with the help of Thermo Mechanical Analyser. The output graphs of all the three sets are shown in Figures 7, 8 and 9.

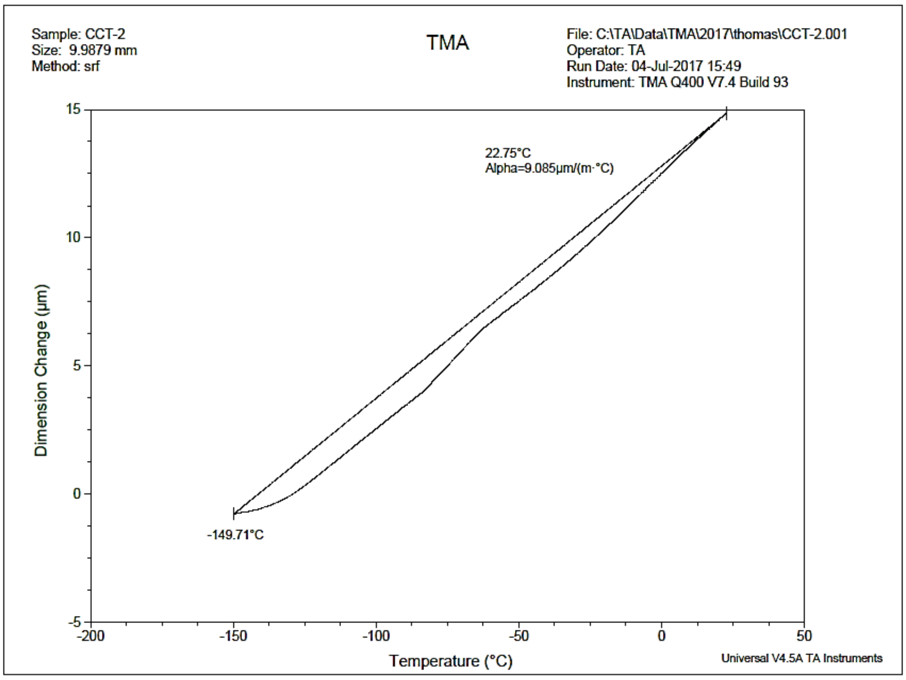

Figure 7. Dimensional changes for untreated specimens

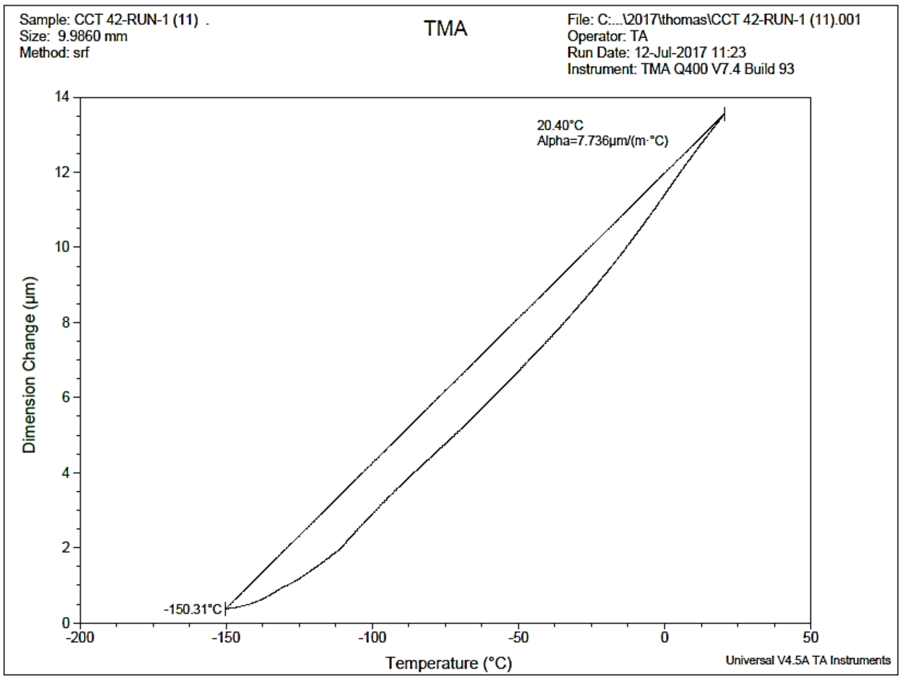

Figure 8. Dimensional changes for cryotreated specimens

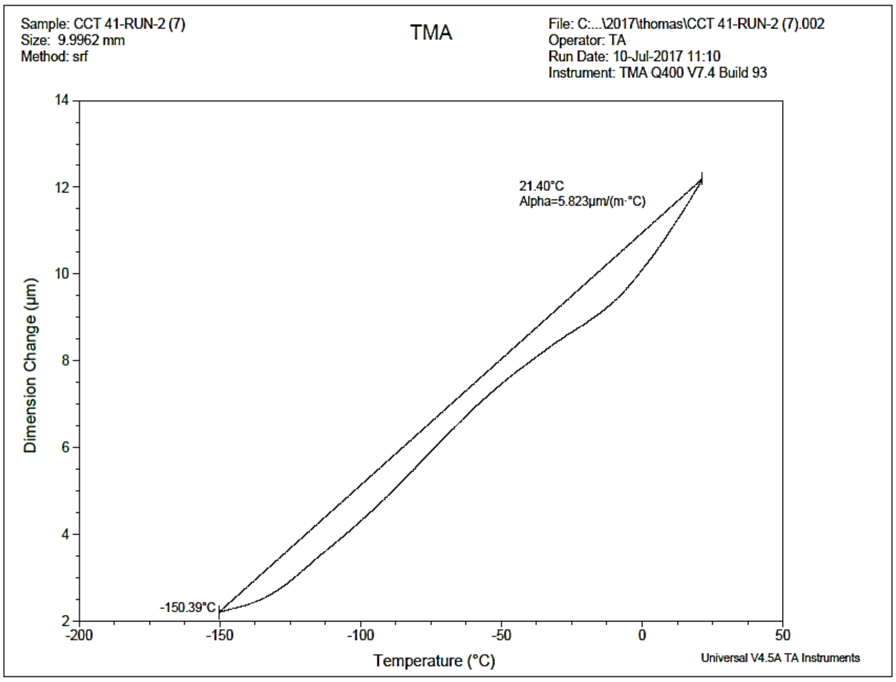

Figure 9. Dimensional changes for cryotreated and tempered specimens

The inbuilt software of the TMA computes the values of coefficients of linear expansions over the entire temperature range at regular intervals of 5K and the mean value is calculated. The experimental results of the calculated α value is shown in Table 1.

Table 1. Coefficients of linear expansion

S. No	Sample	Coefficient of linear expansion ($\mu m/mK$)
1	Untreated	9.172
2	Cryotreated	7.210
3	Cryotreated and tempered	5.823

Untreated specimens exhibited the mean coefficient of linear expansion value of 9.172 $\mu m/mK$ which is higher compared with the other two types of specimens. This is attributed to the presence of residual stresses and soft retained austenite within the material [7]. When the steel specimens are cryotreated, lattice parameters of the atoms change due to the stresses being relieved. Soft, tough and ductile Face Centred Cubic (FCC) structured retained austenite to strong and hard Body Centred Tetragonal (BCT) structured martensite [8, 9].

As observed by Ridvan Gicu et al, during the cryotreatment process, micro sized hard carbide particles are precipitated which fill up the vacancies between the existing larger atoms in the atomic structure[10]. Due to these reasons, the entire atomic structure becomes more compact, hard and strong as compared with the untreated specimens. When the specimens are heated from the cryogenic temperature zone, the expansion of material is hindered both due to the stronger atomic structure and interlocking of the atoms with newly precipitated carbide particles. This effect can be clearly seen by the reduction in coefficient of linear expansion values of the cryotreated specimens (7.210 $\mu m/mK$) and hence increased dimensional stability.

The third case involved the tempering process post cryotreatment. Bensely et al have reported precipitation of still finer nano sized carbide particles known as eta (η) carbides during the tempering process [11]. These rod shaped carbides (approximately 10 nm in diameter and 40 nm in length) further fill up the remaining voids and produce even denser and compact atomic structure This filling effect further enhances the tendency to resist linear expansion when temperature is increase resulting in reduction of the α value to 5.823 μm/mK. This reduced value indicates higher dimensional stability as compared with the previous values.

6. Conclusions

The performance of pressure transducer in space vehicles is very critical and the study of its dimensional stability has its marked significance. In this experimental study, the dimensional stability of the integral diaphragm pressure transducer has been analysed by Thermo Mechanical Analyser. It was possible to increase the dimensional stability of the selected grade of steel by nearly 20% which could be further enhanced by subsequent tempering process. The encouraging results of the study offer scope to try the cryotreatment technique on various engineering materials where dimensional stability is of prime importance.

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