



Thermophysical Properties of Titanium (Ti-6Al-4V) Alloy in the Temperature Range of -125° C to 550° C.

S. Manikandan^{1*}, S. Ramanathan², V. Ramakrishnan³

¹Department of Mechanical Engineering, Annamalai University India

²Department of Manufacturing Engineering, Annamalai University India

³Thermal Systems group, ISRO satellite centre, Bangalore, India

Abstract: Titanium 6Al 4V alloy is an $\alpha + \beta$ alloy, which generally contains alpha and beta stabilizers and is heat treatable to various temperatures. Ti 6Al 4V alloy is designed for high strength at low to moderate temperatures. This alloy is fully heat treated in section sizes up to 15 mm and is used up to approximately 400 °C. Over 70% of all titanium alloy grades melted on a sub grade of Ti 6Al 4V which is used in aerospace, air frame and engine components, The present study is focused on two different types of heat treatments on titanium (Ti 6Al 4V) alloy (solution treated and annealed). The heat treated specimens were thermal cycled to explore the stability and behaviour of these materials for property development. The present study investigates the variation in coefficient of thermal expansion for the heat treated and thermal cycled titanium alloy at various cycles in a newly designed thermal cycling apparatus. In the present investigation Coefficient of thermal expansion of thermal cycled titanium alloy was analyzed using Thermo Mechanical Analyzer (TMA) in the temperature range of 125° C to 550° C. This temperature range gives valuable information for space craft's which undergo thermal cycles from +125°C to 125°C as it moves in and out of the Earth's shadow.

Keywords: Thermal cycling; Coefficient of thermal expansion; Thermo mechanical analyzer; Heat treatment.

Introduction

Ti-6Al-4V titanium alloy is widely used in industrial applications such as aeronautic and aerospace due to its good mechanical properties at high temperatures. The need for lightweight, dimensionally stable materials for critical aerospace applications opened new frontiers of advanced materials. Titanium alloys can withstand the extreme conditions often encountered in space environment [1] Titanium alloys are thermal cycled and tested in TMA. The measurement and characterization of thermo physical properties of titanium alloys such as density, coefficient of thermal expansion (CTE), specific heat (C_p) and thermal conductivity plays an important role. It is essential to evaluate the new material for thermal stability and to evaluate CTE before actual use. Experimentally the CTE can be measured by TMA under the absolute methods. The experiments have been carried out in the temperature range -125° C to 550°C. The measurements of the thermal properties of materials help better understanding of the thermal design [2].

In the near-earth orbit, typical spacecraft encounters natural phenomena such as vacuum, thermal radiation, atomic oxygen, ionizing radiation, and plasma, along with factors such as micrometeoroids and human-made debris. For example, the International Space Station, during its 30-year life, will undergo about

175,000 thermal cycles from +125°C to -125°C as it moves in and out of the Earth's shadow. Re-entry vehicles for Earth and Mars missions may encounter temperatures that exceed 1500°C. Therefore, critical spacecraft mission demands lightweight space structures with high pointing accuracy and dimensional stability in the presence of dynamic and thermal disturbances.

Thermal cycling

Thermal cycling is a temperature modulation process developed to improve the performance, strength and longevity of a variety of materials. Thermal cycling has been applied chiefly to metals to-date, although the process is also beneficial to Ti-6Al-4V, It is currently used by a number of industries where enhanced material performance is desired.

During the thermal cycling process, materials are alternately cooled and heated until they experience molecular reorganization. This reorganization "tightens" or optimizes the particulate structure of the material throughout, relieving stresses, and making the metal denser and more uniform (thereby minimizing flaws or imperfections). The tighter structure also enhances the energy conductivity and heat distribution characteristics of the material. Thermal Cycling minimizes "hotspots", enhances cooling and impedes the ability and tendency of metals to vibrate. Significantly reducing vibration as a factor in metal fatigue slows down the metal's eventual failure or breakage. Corrosion resistance is enhanced as a result of molecular uniformity because the metal's ability to impede oxidation and chemical degradation is strengthened. [3]

Ti-6Al-4V alloys considered to be potential candidate materials for many space applications. Titanium can withstand the extreme conditions often encountered in space environment [4]. Critical spacecraft missions require dimensional stability in the presence of varying thermal conditions.

The ASTM E 831 has approved the CTE measurement by TMA [5]. This approach is simple, fast, fairly accurate and cost effective. The CTE experiments have been carried out in the temperature range -125°C to 550°C. This study is concerned with Thermophysical aspects of titanium alloys with a view to explore possible space applications.

Experimental Study

The material was purchased from Mishra Dhatu Nigam Limited (A Government of India Enterprise, Hyderabad) at Hot rolled condition. The cylindrical specimen of dimensions 5.0 ± 0.1 mm in diameter and a length of 7.5 ± 0.1 mm were machined from the CNC machine. The specimens were subject to two different types of heat treatments (Solution treated and annealed).The heat treated specimens were thermal cycled in a specially designed thermal cycling apparatus to explore the stability and behaviour of these materials for property development. The chemical composition of titanium alloy used in the present investigation is given in the Table.1.

Table (1) Composition of Ti-6Al-4V alloy

Element	C	Fe	N ₂	O ₂	Al	V	Ti
Wt %	<0.08	<0.25	<0.05	<0.2	5.5-6.7	3.5-4.5	Balance

Heat treatment of Ti-6Al-4V alloy

Annealing is the method of heat treatment in which, the test specimens were heat treated to 750°C for 4 hours and quenched in atmospheric air. Annealing increases strength and ductility. In solution treatment, the test specimens were heat treated to 950°C for 1 hour and quenched in caustic soda for 10 minutes with a quench delay of 6 seconds. After completing the solutionizing, the same specimens were treated to aging for 450°C for a period of time 4 hours. After the heat treatment the specimens were quenched in the atmospheric air. Aging increases the strength and hardness [6, 7].

Fabrication of thermal cycling apparatus

The basic thermal cycling apparatus comprises a muffle furnace, forced air cooling unit, electronic experimental controls unit and timer unit. The timer unit controls the heating and cooling time period during thermal cycling process and regulates the flow of compressed air to the pneumatic cylinder through the electrically operated pneumatic valve. Further the experimental set-up consist of pneumatic components like double acting reciprocating air compressor, air control main valve, air filter, air regulator, air lubricator, electrically operated pneumatic valve and pneumatic cylinder ,which are shown in Fig.1.and the Photographic views of thermal cycling apparatus is shown in Fig. 2.

The pneumatic cylinder piston carries the cage, in which the test sample is placed. The cage is made up of stainless steel with three compartments to carry similar test samples in it. The timer unit counts the number of cycles and stops the process after completing the required number of cycles. Manual handling and monitoring of the sample during the thermal cycling process is minimized due to this automation. Pneumatic piston actuator was built to cycle the specimen in and out of the furnace. Where, the required temperature was maintained in the furnace. The thermal cycle imposed had dwell time of 2 minutes in and out of the furnace. Specimens were cooled with atmospheric air in order to avoid the brittleness and increase the ultimate strength. Forced air cooling was used to cool the thermal cycled specimen. Air at a pressure of 0.2 N/mm² was sprayed to cool the specimen for 2 minute to attain atmospheric temperature. Heat treated specimens were subjected to thermal cycling in the range of 250 to 1500 cycles.

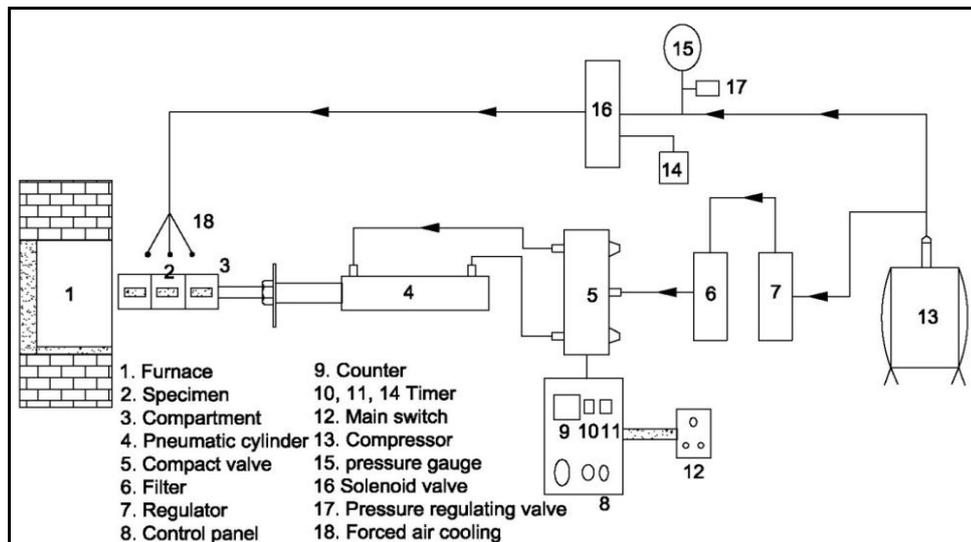


Fig. (1) Line diagram of thermal cycling experimental setup



Fig . (2) Photographic views of thermal cycling apparatus

The measurement of CTE requires an approach that employs both length and temperature measurement simultaneously. All CTE measurements were carried out in the research grade Thermo Mechanical Analyzer TMA Model Q400 V7.4 Build 93 (TA Instruments, USA) in ISRO Satellite Centre, Bangalore. It measures the vertical displacement in the test specimen and the first derivative of the displacement as a function of the temperature. All the experimental runs were carried out in the temperature range of -125°C to 550°C . The heating rate selected for all measurements was $10^{\circ}\text{C}/\text{min}$ and was continuously monitored and controlled by the computer based data acquisition system. The resulting thermo gram is the trace of the (probe displacement) dimensional change in $\mu\text{m}/\text{m}$ versus the sample temperature ($^{\circ}\text{C}$) [8]. In all cases measurements were made with a minimum of three samples (1, 2 and 3). The measurement on each sample was carried out for three times. Instead of pre-conditioning the samples in a separate furnace the first run carried out inside the module helped to remove the process induced residual stresses, and the surface absorbed moisture. The second run carried out after cooling the sample to room temperature without disturbing the experimental set-up provided realistic values. In all cases the measured values obtained in the second run were better and reliable.

The precision and accuracy of determining the CTE depends upon the simultaneous measurement of temperature and the change in length [9]. The systematic errors can be reduced by good up keeping and careful calibration of the system. Repeat measurements confirm the precision of the measurement data. The individual contributions of errors were worked out and the total measurement error in CTE measurement by TMA is restricted to $\pm 2\%$ by adopting a well-laid systematic measurement approach.

Results and Discussion

The thermal cycled specimens were tested in TMA. The CTE measurements were carried out on each three samples. Measurements were carried out in all six samples (500, 1000 & 1500 cycles for Annealed and Solution treated) and a total experiment of 54 runs were carried out for the measurement of Coefficient of thermal expansion & Dimension change. Samples were kept in a separate furnace, the first run carried out inside the module helped to remove the process induced residual stresses and the surface absorbed moisture. The second run was carried out after cooling the sample at room temperature, without disturbing the experimental set up, provided residual stresses. In all the cases second run was better and reliable. The heating rate selected for all measurements was $10^{\circ}\text{C}/\text{min}$. All the experimental runs were carried out in the temperature range -125°C to 550°C . Nitrogen was used as a purge gas at the flow rate of $50.0\text{ ml}/\text{min}$. A small static force of (0.05 N) was applied on the sample by the measuring probe. The TMA traces the Dimension change (DC) and Co efficient of thermal expansion (CTE) as a function of temperature.

When a metal is alternately subjected to heating and cooling cycles, it must maintain a certain tolerance of dimensions and a low coefficient of thermal expansion is desirable. When in contact with a metal of a different coefficient, this consideration assumes greater importance. Titanium has a low coefficient of thermal expansion when compare to stainless steel, copper and aluminium. From TMA it is observed that Co efficient of thermal expansion (CTE) increases with increase in temperature. But the increase is not linear, retardation of the curve occurs in the negative temperature range and it is regained after attaining in the positive temperature (-75°C to 100°C) which is witnessed from figure 3 and 4. Retardation of curves in negative temperature occurs only in lower number of thermal cycles (500 cycles). This retardation nature gets diminished in higher number of thermal cycles (1500 cycles) due to molecular reorganization. This reorganization "tightens" or optimizes the particulate structure of the material throughout, relieving stresses, and making the metal denser and more uniform.

Figs.3 to 11 represents the variation of Co efficient of thermal expansion of the Annealed (ACTE) & Solution treated (SCTE) specimens at 500, 1000, 1500 cycles, High Co efficient of thermal expansion is obtained at 1500 cycles. Although high Co efficient of thermal expansion is not desired for good materials, titanium is comparatively very low with Aluminium alloys and Aluminium composites. Early experiments conducted by Karthikeyan et al.[10] shows that Aluminium alloy and Aluminium Si c_p composites shows up to $48\ \mu\text{m}/\text{m}^{\circ}\text{C}$ at 500°C is very high when compared with Ti-6Al-4v value of $10.8\ \mu\text{m}/\text{m}^{\circ}\text{C}$.

From Figs 3 to 11 it is observed that in both Annealed and solution treated specimen, at 500 cycles, there is a retardation in the temperature range of -75°C to 100°C , this occur due to sudden change from ultralow temperature to positive side. retardation occurs at 100°C to 200°C at 500 cycles, another retardation

occurs at 300°C to 400°C at 1000 & 1500 cycles due to Marten site to $\alpha + \beta$, where Martensite is less dense when compare to β phase which is proved by Homprova et al.[11] in Dynamic phase evolution of Titanium alloys. In all the cycles (500, 1000, 1500) the expansion of solution treated specimen is higher than Annealed specimen.

As per various literature survey, Co efficient of thermal expansion of Ti-6Al-4v is of 8.6, 9.2 & 9.7 $\mu\text{m}/\text{m}^\circ\text{C}$ at 20, 200 & 500 °C before heat treatment and thermal cycling , After heat treatment and thermal cycling there is only marginal rise of CTE which shows at an average value of 8.4, 9.5 & 10.3 $\mu\text{m}/\text{m}^\circ\text{C}$ at 20, 200 & 500° C. CTE value gets decreases at lower temperature of 20° C because of a sudden temperature change from negative side to positive side during the measurement.

Figs. 3, 4 & 5 represents the variation of co-efficient of thermal expansion of the two heat treated (Annealed & Solution treated) specimens at 500, 1000, 1500 cycles respectively, High Co efficient of thermal expansion is for Solution treated specimens. Retardation occurs at ultra low temperature in 500 & 1000 cycles, a uniform curve without any retardation was obtained at 1500 cycles due to molecular reorganization is witnessed.

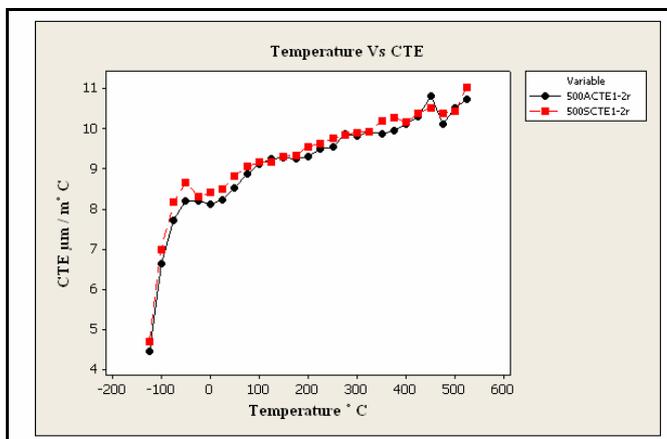


Fig. (3) CTE of Annealed and Solution treated for 500 thermal cycled Ti-6Al-4V alloy ACTE, SCTE)

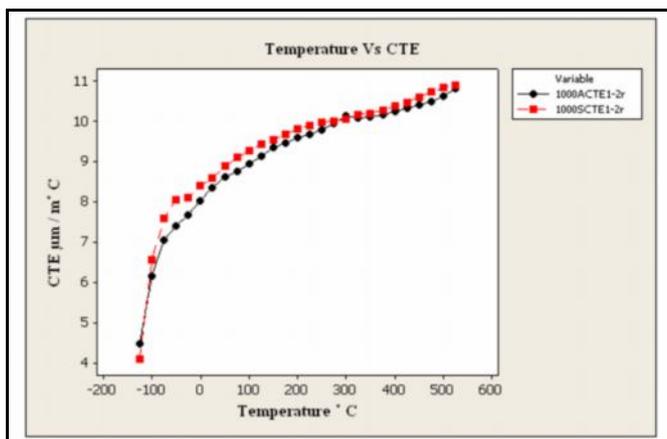


Fig. (4) CTE of Annealed and Solution treated for 1000 thermal cycled Ti-6Al-4V alloy (ACTE, SCTE)

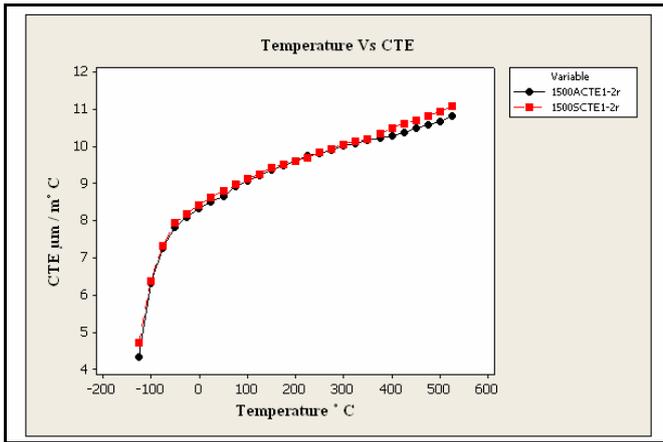


Fig. 5. CTE of Annealed and Solution treated for 1500 thermal cycled Ti-6Al-4V alloy (ACTE, SCTE)

Figs. 6, 7 & 8 shows the comparative values of Co efficient of thermal expansion of the Annealed specimens at 500, 1000, 1500 cycles, for the samples tested for 2nd run. Although CTE value of the thermal cycled specimens were slightly higher than the without heat treated and thermal cycled specimens, there is no retardation occurs in the curve. This indicates thermal cycling relieves the internal stresses, and makes the alloy free from deformation and failure behavior to considerable extent. The thermal property depends on the particulate size, shape and distribution in the alloy. For a good heat material, it is desirable to have low CTE and Cp property. CTE values observed through TMA is very less when compared to Aluminium alloy and Aluminium SiCp composites at all these temperature range. Hence Ti-6Al-4V alloy is a good heat material, which has low CTE property after heat treatment and thermal cycling.

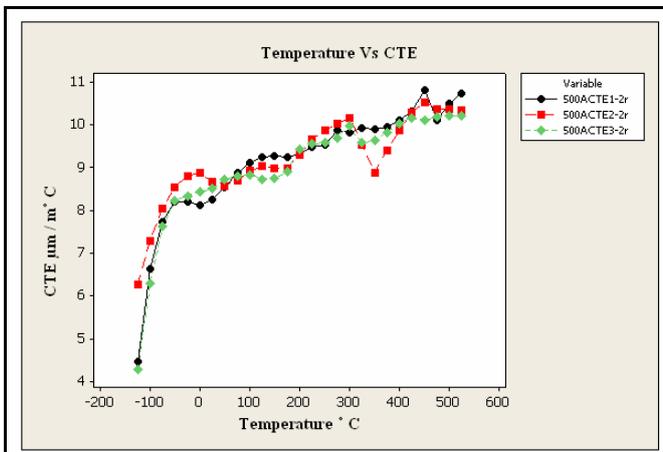


Fig. 6. CTE of Annealed & 500 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (ACTE)

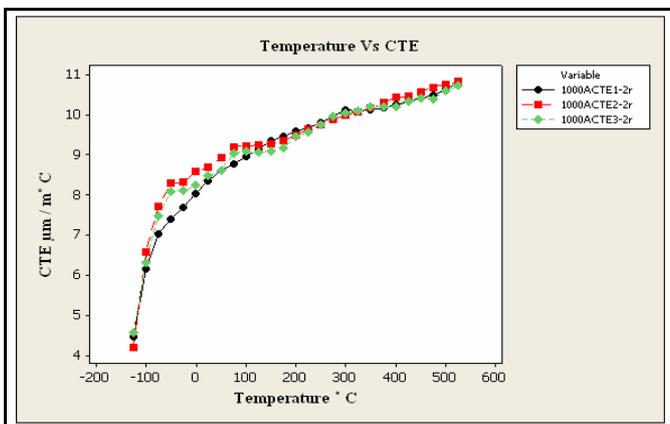


Fig. 7. CTE of Annealed & 1000 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (ACTE)

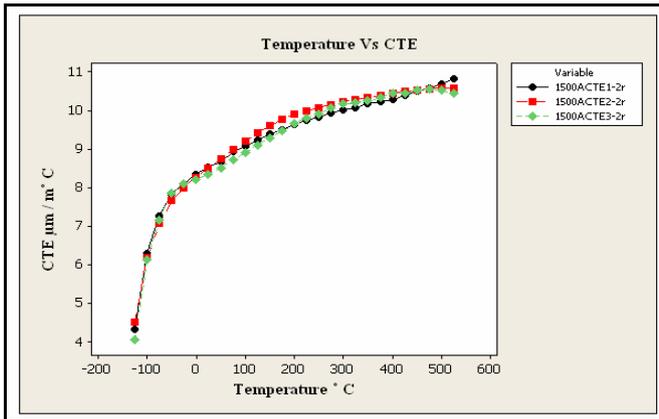


Fig. 8. CTE of Annealed & 1500 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (ACTE)

Figs. 9, 10 & 11 shows the comparative values of coefficient of thermal expansion of the Solution treated specimens at 500, 1000, 1500 cycles, for the samples tested for 2nd run. First run carried out inside the module which helps to remove the process-induced residual stresses and the surface absorbed moisture. The second run carried out, after cooling the sample to room temperature, without disturbing the experimental set-up, provided realistic values. Second run shows the peak values in all the three figures. In Fig. 9, retardation occurs in the negative temperature as well as in the 500° C range, and this is due to the presence of residual stresses, which is partially present in lower cycles. And a smooth flow of curve obtained in higher cycles is due to relieve of residual stress and molecular re organization.

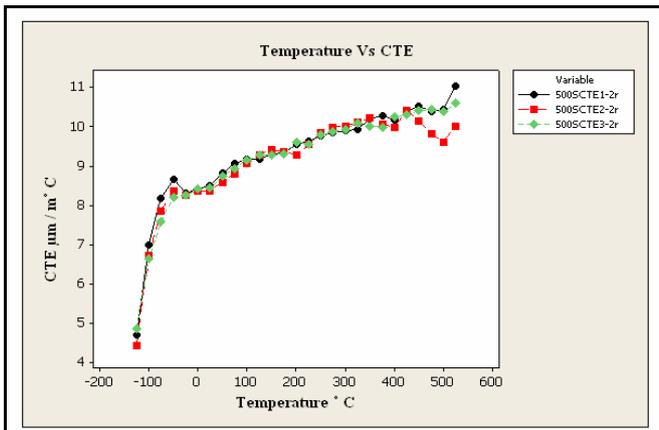


Fig. 9. CTE of Solution treated & 500 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (SCTE)

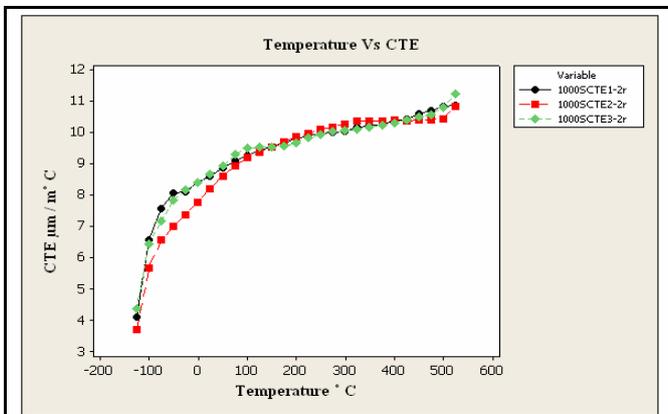


Fig. 10. CTE of Solution treated & 1000 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (SCTE)

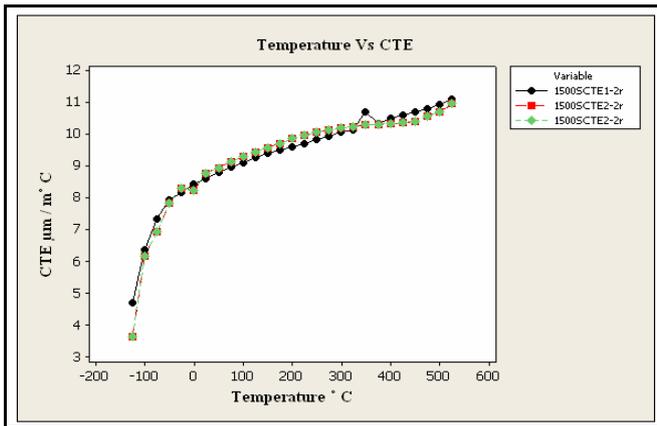


Fig. 1. CTE of Solution treated & 1500 thermal cycled Ti-6Al-4V alloy for Samples 1, 2 & 3 – 2run (SCTE)

From Figs. 12, 13, & 14 it is observed that there is no marginal variation in Dimension change in Annealed & Solution treated specimens in all the cycles. Dimension change measured in all the samples increases with increase in temperature. Increase of Dimension change with respect to temperature is linear in all the samples and for the entire test run. And there is a slight deviation occurs in the second run when compared to first run in all the samples.

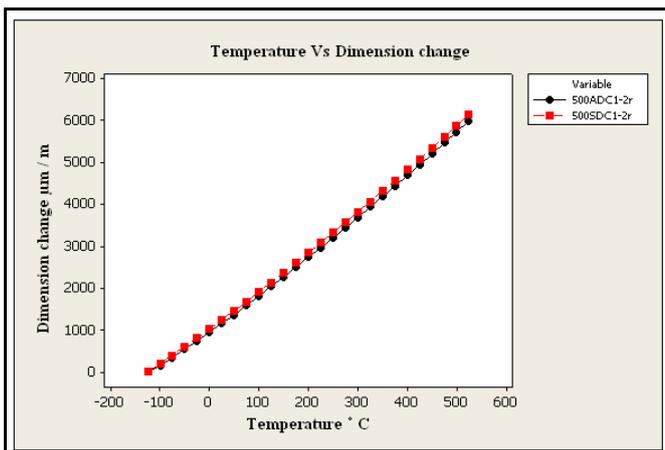


Fig. 2. Dimension change of Annealed & Solution treated Ti-6Al-4V alloy for 500 thermal cycles

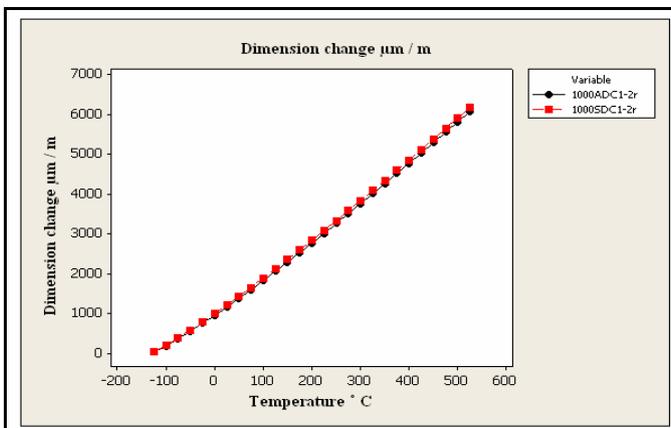


Fig. (13) Dimension changes of Annealed & Solution treated Ti-6Al-4V alloy for 1000 thermal cycles

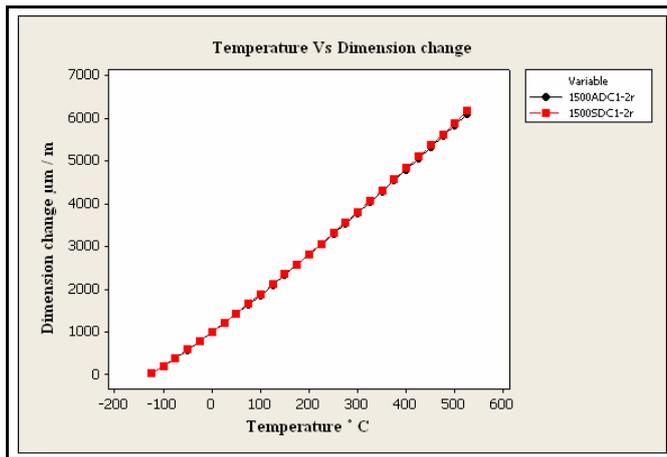


Fig.(14) Dimension changes of Annealed & Solution treated Ti-6Al-4V alloy for 1500 thermal cycles

In the annealed specimen CTE at lower cycle (500 cycles) is higher up to 100°C, after 100°C CTE value decreases considerably at higher temperature, (100°C to 525°C) Thermal cycling influence the CTE value at 1000 & 1500 cycles in annealed specimen.

Thermal cycling does not influence the CTE value after 1000 cycles in solution treated specimen. Thermal cycling influences the CTE value without any retardation at 1500 cycles. Another important characteristic of titanium-base materials is the reversible transformation of the crystal structure from alpha (hexagonal close-packed) structure to beta (body-centered cubic) structure when the temperatures exceed certain level. This allotropic behaviour, which depends on the type and amount of alloy contents, allows complex variations in microstructure and more diverse strengthening opportunities than those of other nonferrous alloys such as copper or aluminum [12,13&14].

Conclusions

From this investigation, it is observed that co-efficient of thermal expansion increases with increase in temperature. But the increase is not linear, retardation of the curve occurs in the negative temperature range and it is regained after attaining in the positive temperature (-75 °C to 100 °C) Retardation of curves in negative temperature occurs only in lower number of thermal cycles (500 cycles). This retardation nature gets diminished in higher number of thermal cycles (1500 cycles) due to molecular reorganization. This reorganization "tightens" or optimizes the particulate structure of the material throughout, relieving stresses, and making the metal denser and more uniform.

The following conclusions were made from the experimental study.

- Both solution treated and Annealed specimen, there is retardation in the temperature range of -20°C to 100°C, at 500 & 1000 cycles, which is due to sudden change from ultralow temperature to positive side.
- Retardation occurs between 100°C-200°C at 500 cycles and between 300°C-400°C at 1000 & 1500 cycles. In all the cycles (500, 1000, and 1500) the expansion of solution treated specimen is higher than Annealed specimen.
- There is no retardation occurs in the temperature range of -20°C to 100°C at 1500 cycles, thermal cycling influences more uniform curves at higher cycles.
- Coefficient of thermal expansion is higher for Solution treated specimens. Retardation occurs at ultra low temperature in 500 & 1000 cycles, a uniform curve without any retardation was obtained at 1500 cycles.
- Thermal cycling does not influence the CTE value at 1000 cycles in solution treated specimen.
- There is no marginal variation in Dimension change in Annealed & Solution treated specimens in all the cycles.
- Increase of Dimension change with respect to temperature is linear in all the samples and for the entire test run

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