CHAPTER 4

RESULTS AND DISCUSSION

Ti6Al4V alloy and Ti6Al4V alloy and with different weight percents of (10, 15 and 20%) Nano-SiCp (Ti6Al4V/Nano-SiCp Composites) specimens were fabricated by powder metallurgy technique. Thermal analysis technique such as specific heat property and coefficient of thermal expansion (CTE) on the specimen are calculated to explore thermal characteristics of the materials. The favorable and instable conditions such as ranges of temperature of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites have been characterized, analyzed, simulated, and suggested[157-159].

4.1 CHARACTERIZATION OF NANO SILICON CARBIDE (Nano- SiCp)

The pulverized silicon carbide powder samples were characterized by Atomic force microscope (AFM) to analyze the topography and by Particle analyzer (Zetasizer). In this work, experiments were conducted to compare the silicon carbide powder, roughness value, particle size reduction and agglomeration by varying the Ball to Powder (BP) ratio of ball milling parameters.[74]

The fast reduction in particle size and the agglomeration effect was confirmed by SEM photographs of, as received powder image Figure 4.1, and the milled powder images of 8 hrs with BP ratio 20:1 shown in Figure 4.2(a) & 8 hrs with BP ratio 40:1 shown in Figure 4.2(b). This confirms the generation of agglomeration as particles becomes smaller. Another possibility that had to be considered was contamination that could occur in the processes, namely, the increase in surface area could be the result of breaking of SiC particles plus the addition of other small particles from the milling media.[74]
Fig 4.1 SEM image of as received SiC powder

Fig 4.2(a) SEM image of sample1
(20:1 BP ratio)

Fig 4.2(b) SEM image of sample2 (40:1BP ratio)
4.1.1 Atomic Force Microscopy (AFM)

For better understanding of the morphology of a surface, a quantitative description of the surface topography must be carried out. The topography matrix data should be treated in each profile line (2D) or over all profiles extending the analysis to surface (3D). The amplitude parameters are the principal parameters in characterizing the surface topography [76]. The average roughness (Ra) is the most used amplitude parameter and was measured. AFM images of both samples in two dimensional formats are shown in Figures 4.3(a) & 4.3(b). Particle distribution found in the 10×10 μm area is explored by drawing a line profile drawn across the 2D image at 6.5 μm & 5.25 μm is represented as red line and a line drawn at 5.25 μm & 5.75 μm is indicated by the green line respectively. The average surface roughness (Rₚ) was found as 1.277 nm and 1.485 nm for both samples respectively. An AFM image of SiCp displayed in a 3-D format is shown in Figures 4.4(a) & 4.4(b). These give a rendition of what the surface topography actually looks like. It is observed that there is no much change in roughness value which represents; roughness is independent of BP ratio.

Fig 4.3(a) Particle distribution of sample-1 in 2D format AFM image
Fig 4.3(b) Particle distribution of sample-2 in 2D format AFM image

Fig 4.4(a) Particle distribution of sample-1 in 3D format AFM image
4.1.2 Particle Analyzer (Zetasizer)

The results of particle analyzer are shown in the Figures 4.5(a) & 4.5(b). It is observed that, the intensity of nano particles higher in the range between 100 nm to 1000 nm. From the Figure 4.5(a) it is observed that, the average particle size of 432.9 nm obtained with the parameters of milling time 8 hrs and BP ratio of 20:1. From the Figure 4.5(b) it is observed that, the average particle size of 315.5 nm obtained with the parameters of milling time 8 hrs and BP ratio of 40:1. The results reveal that by increasing the BP ratio, it considerably reduces the particle sizes.[72-74] When comparing the particle size statistics report by intensities of both sample, shown in the Figures 4.6 (a) & 4.6 (b), it is observed that in the case of 20:1 BP ratio, the intensity of nano particles is varied from the range of 100 nm to 8000 nm but in the case of 40:1 BP ratio, the intensity of particles varied in the range of 100 nm to 1000 nm only. So, the results obtained from the ball milling parameters of milling time 8 hrs and BP ratio 40:1 are narrow and superior.[74]
### Size Distribution Report by Intensity

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#### Sample Details
- **Sample Name**: SiC 1
- **SOP Name**: Krishnamohan EGSPC.sop
- **General Notes**:
  - **File Name**: Installation.dat
  - **Record Number**: 443
  - **Material RI**: 1.59
  - **Material Absorption**: 0.01
- **Dispersant Name**: Water
- **Dispersant RI**: 1.330
- **Viscosity (cP)**: 0.8872
- **Measurement Date and Time**: Saturday, October 01, 2011

#### System
- **Temperature (°C)**: 25.0
- **Duration Used (s)**: 80
- **Count Rate (kcps)**: 119.8
- **Measurement Position (mm)**: 4.85
- **Cell Description**: Disposable sizing cuvette
- **Attenuator**: 0

#### Results

<table>
<thead>
<tr>
<th>Diameter (nm)</th>
<th>% Intensity</th>
<th>Width (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Z-Average (d.nm):</strong> 432.0</td>
<td>Peak 1: 438.0</td>
<td>84.6</td>
</tr>
<tr>
<td><strong>Pdl: 0.262</strong></td>
<td>Peak 2: 3033</td>
<td>15.4</td>
</tr>
<tr>
<td><strong>Intercept: 0.778</strong></td>
<td>Peak 3: 0.000</td>
<td>0.0</td>
</tr>
</tbody>
</table>

**Result quality**: Good

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Fig 4.5 (a) Average particle size of sample-1
Fig 4.5 (b) Average particle size of sample-2
Fig 4.6 (a) Particle size statistics by intensity of sample-1
Fig 4.6 (b) Particle size statistics by intensity of sample-2
It was observed that there is no much change in roughness value of both samples and it is inferred that roughness value is independent of BP ratio. Ball to powder ratio of 20:1 parameter reduce the particle size to an average of 432.9 nm and Ball to powder ratio of 40:1 parameter reduce the particle size to an average of 315.5 nm. It is observed that by increasing the BP ratio, the particle size can be considerably reduced [72]. Particle size is directly proportional to the BP ratio of ball milling parameter[74].

Hence, the ball to powder ratio of 40:1 has been considered for nano silicon carbide powder preparation (Nano-SiCp).

4.2 THERMAL ANALYSIS

Thermal analysis technique such as specific heat property and co-efficient of thermal expansion (CTE) on Ti6Al4V alloy and Ti6Al4V Nano-SiCp specimens are calculated to explore thermal characteristics of the materials. The favorable and unstable conditions such as ranges of temperature of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites have been characterized, analyzed, simulated and suggested.[132,139]

The thermal characteristics of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites are studied. The specific heats of materials have been measured in a temperature range of -100°C to 375°C in steps of 25°C. Similarly, the coefficients of thermal expansion of materials have been measured in a temperature range of -100°C to 400°C in steps of 25°C. The favorable and unstable temperature range of materials have been characterized, analyzed and represented in graphs. These findings can be easily used in industries for selection and design of effective thermal control systems for space crafts, etc. The results of the individual thermal characteristic analysis for different materials are discussed in the following sections separately; summary based on above analysis is given at the end of the discussion.[132, 139]
4.2.1 SEM Micro Graph of Test Specimens

The initial microstructure of Ti6Al4V alloy is shown in Figure 4.7. The microstructure of Ti6Al4V alloy with different volume percents of nano-SiCp (10, 15 and 20%) composites are shown in Figures 4.8 - 4.10.

Fig. 4.7 SEM micrograph of Ti6Al4V alloy

Fig. 4.8 SEM micrograph of Ti6Al4V /10% Nano-SiCp

Fig. 4.9 SEM micrograph of Ti6Al4V /15% Nano-SiCp

Fig. 4.10 SEM micrograph of Ti6Al4V / 20% Nano-SiCp
4.2.2 X-ray Diffraction (XRD)

The X-ray Diffraction (XRD) analysis is carried out to identify the composition of the Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites and conforming of the main elements of composites. The major peaks and minor peaks of intermetallic phases such as Ti, Al, V and nano-SiCp are found in the composites. X-ray Diffraction (XRD) plot of Ti6Al4V alloy with different volume fractions of nano-SiCp (10, 15 and 20%) composites are shown in Figures 4.11-4.14.

![Fig. 4.11 X-ray diffraction (XRD) plot of Ti6Al4V alloy](image1)

![Fig. 4.12 X-ray diffraction (XRD) plot of Ti6Al4V alloy/10% Nano-SiCp](image2)

![Fig. 4.13 X-ray diffraction (XRD) plot of Ti6Al4V alloy/15% Nano-SiCp](image3)

![Fig. 4.14 X-ray diffraction (XRD) plot of Ti6Al4V alloy/20% Nano-SiCp](image4)
4.3 STUDY OF SPECIFIC HEAT PROPERTY OF Ti6Al4V ALLOY AND Ti6Al4V/Nano-SiCp COMPOSITES USING DIFFERENTIAL SCANNING CALORIMETER (DSC)

Differential scanning calorimetry (DSC) is the most common dynamic technique used in calorimetry and thermal analysis. In this approach, the heat flow rate associated with a thermal event which can be measured as a function of time and temperature to obtain quantitative information about heat capacities or phase transitions [133]. DSC measurements are generally affected by a number of factors which includes, the sample features (geometry, particle size, mass, thermal conductivity, sample-pan contact surface) and the experimental conditions (scanning rate, atmosphere).

The heat capacity measurements were carried out on each two samples of Ti6Al4V alloy and Ti6Al4V with different volume percents of nano-SiCp (10, 15, and 20%) composites. Measurements were carried out thrice in all eight samples. The heat capacity measurements of the sample materials were coded as shown in the Table 3.4. Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites have been developed indigenously and tested in Differential Scanning Calorimetry (DSC).

Instead of preconditioning the samples in a separate furnace, the first run carried out inside the module which helps to remove the process-induced residual stresses and the surface absorbed moisture. The second run and third 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 and third run were better, repeatable, reproducible and reliable. The heating rate selected was ramp 20°C/min to 375°C and isothermal for 10 minutes. Helium was used as a purge gas at the flow rate of 25 ml/min.[132] The DSC traces of the heat capacity as a function of the temperature are plotted Figures 4.15-4.22.

In analyzing linear heating experiments, the baseline of the DSC needs to be carefully considered because the baselines will generally be temperature and time dependent [90]. In heat capacity measurement of a material using DSC, a sample of the material to be investigated is placed in the DSC with a reference of known heat capacity \(C_{p,\text{ref}}(T)\). The heat capacity of the sample can then be determined from heat flow \(q\) from the DSC as
\[ C_{p, \text{sample}}(T) = q\beta + C_{p,\text{ref}}(T) \quad (4.1) \]

where \( \beta \) is the heating rate. As heat capacities of many substances are well known, the determination of heat capacity can also be used to calibrate the heat flow measurement in the DSC [133].

### 4.3.1 Scanning Run Effect

The heat capacities of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites were measured. The interaction between the matrix and reinforcement will also influence the resultant heat capacities of the composites. The results of the heat capacity measurement graphs are shown in Figures 4.15-4.22 as a function of the temperature.

From Figures 4.15-4.22, it is observed that, in the entire Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites, variation found in the measured heat capacity between the first, second and third runs. This is due to enthalpy relaxation effect due to stress induced by processing and the surface absorbed moisture depending on complexity and alloying content [90].

It is observed that the heat capacity of the samples increases with an increase in temperature. But the increase is not perfectly linear and this can be attributed to an increase in the amplitude of vibration of the atoms of the constituents of the new material with increase in temperature. There is a uniform rise in heat capacity from ultra low temperature to 75°C. The heat capacity measurement of alloy and composites do not show any exothermic and endothermic peaks at same temperature values. This phenomenon is due to the higher melting points of Titanium alloy and composites [131]
Fig. 4.15 Heat capacity of run effect of Ti6Al4V alloy (Sample-1)

Fig. 4.16 Heat capacity of run effect of Ti6Al4V alloy (Sample-2)
Fig. 4.17 Heat capacity of run effect of Ti6Al4V/10%Nano-SiCp composite (Sample-1)

Fig. 4.18 Heat capacity of run effect of Ti6Al4V/10%Nano-SiCp composite (Sample-2)
Fig. 4.19 Heat capacity of run effect of Ti6Al4V/15%Nano-SiCp composite (Sample-1)

Fig. 4.20 Heat capacity of run effect of Ti6Al4V/15%Nano-SiCp composite (Sample-2)
Fig. 4.21 Heat capacity of run effect of Ti6Al4V/20%Nano-SiCp composite (Sample-1)

Fig. 4.22 Heat capacity of run effect of Ti6Al4V/20%Nano-SiCp composite (Sample-2)

It is observed that in the second and third runs heat capacities are nearly same when compared with first run. This is because of removal of the process-induced residual stresses.
and the surface absorbed moisture in the specimen during the first run. The specific heat, however, shows a large increase versus temperature below room temperature. This can be explained by the Debye theory [135], which describes the temperature dependence of the specific heat. At low temperatures, the specific heat should increase proportionally to $T^3$. At high temperatures, the specific heat should converge to a constant value. The temperature range in which the material starts to differ from the $T^3$ depends on the Debye temperature, which is a material-specific property. This temperature is comparably high for carbon materials (above 2000 K). Therefore, one can expect that the specific heat below and around room temperature still closely resembles the $T^3$ behavior [136].

### 4.3.2 Effect of Addition of Different Volume Fraction of Nano-SiCp with Ti6Al4V Alloy in Heat Capacity Measurement

The heat capacity of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites were measured. The interaction between the matrix and reinforcement will also influence the resultant heat capacities of the composites. From Figures 4.23 to 4.28, it is observed that with increasing the volume fraction percentage of the reinforcement material (nano-SiCp), the heat capacity of the MMCs decreases in all the runs. Though the heat capacity of all the MMCs increased with an increase in temperature and with increase in nano-SiCp content, the heat capacity also decreases for any given measured temperature range.

From Figures 4.23 to 4.28 it is observed that alloy heat capacity values are increasing proportionally with temperature, but it is not so in all the composites due to the presence of nano-SiCp, and also it is observed that the heat capacities of all composites are less than the alloy, due to the presence of reinforcement materials. In all the runs Ti6Al4V/15% Nano-SiCp composites and Ti6Al4V/20% Nano-SiCp composites heat capacity values are more or less same but there is no consistency and repeatability. But in the case of Ti6Al4V/10% Nano-SiCp composite heat capacity values are more consistent and repeatable in all runs.

It is observed that $C_p$ values of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites significantly varies under the identical conditions. Heat capacities of Ti6Al4V/Nano SiCp composites are less than the Ti6Al4V alloy, which is due the fluctuations in the trend indicate the misfit strains introduced in the MMCs by the particulate reinforcement and reduction in the interface area compared to a uniform geometry[132].

The heat capacities of matrix and reinforcement are different, because of this phenomenon; the heat capacities of the Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V Ti alloy material
From figures 4.15 – 4.22 it is observed that the heat capacity of the samples increases with an increase in temperature. The increase is perfectly linear form ultra low temperature to 75°C. This region is stable, but beyond 75°C the increase is not perfectly linear, this region is unstable region.

![DSC – Temperature Vs Heat capacity](image)

Fig. 4.23 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 1st run)
Fig. 4.24 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 2nd run)

Fig. 4.25 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 3rd run)

From Figure 4.25, it is observed that, the variation found in the measured heat capacity between Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composite materials (for first
specimens during third run). The heat capacities of composite materials are less than the alloy. The Ti6Al4V/15%Nano-SiCp and Ti6Al4V/20%Nano-SiCp composites show high heat capacity than Ti6Al4V/10%Nano-SiCp composite in all runs. Further, the Ti6Al4V/15%Nano-SiCp composites show higher heat capacities when compared with Ti6Al4V/20%Nano-SiCp composites. But both composites show almost similar heat capacity values with inconsistency. The heat capacities of matrix and reinforcement are different, because of this phenomenon; the heat capacities of the Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy material.

Fig. 4.26 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 1st run)
Fig. 4.27 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites
(2\textsuperscript{nd} sample in 2\textsuperscript{nd} run)

From Figure 4.27, it is observed that the variation also found in the measured heat
capacity between Ti6Al4V alloy and composite materials for second specimens during second
run. The heat capacities of composite materials are less than the alloy. The
Ti6Al4V/10\%Nano-SiCp composites show less heat capacity than Ti6Al4V/15\% Nano-SiCp
and Ti6Al4V/20\%Nano-SiCp composite during second run. Further, all the composites show
almost uniform change in heat capacity values with respect to temperatures. The heat
capacities of matrix and reinforcement are different, because of this phenomenon; the heat
capacities of the Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy material.
Fig. 4.28 Heat capacities of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 3rd run)

From Figure 4.28, it is observed that, the heat capacity of the all composites less than the alloy during third run. All the measured values trends are in similar pattern. The variations in heat capacity of alloy and all composites are due to the presence of the nano-SiCp in the composites. The presence of the nano-SiCp reduces the heat capacity with increase in its volume percentages. But, the heat capacities of 10% composite are very less in the measured temperature range. The heat capacity measurement of composites shows, similar pattern at same temperature values. Heat capacities of Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy.

From Figures 4.25 & 4.28 It is observed that the heat capacity values of Ti6Al4V/15%Nano-SiCp composite and Ti6Al4V/20%Nano-SiCp composite are nearly similar in first sample but huge difference in second sample and there is no consistency is observed. It is also observed that heat capacity of Ti6Al4V alloy and Ti6Al4V/10%Nano-SiCp composites show similar trends in both samples.

The specific heat increases with increase in temperature as it described in the Debye theory [135]. The heat capacities of all composites are less than the heat capacity of the alloy in second run on-wards. All the measured values trends are in similar pattern. The variations in heat capacity of alloy and all composites are due to the presence of the nano-SiCp in the
composites. Heat capacities of Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy. During the heat capacity measurement, alloy and composites shows, similar patterns at same temperature values. The heat capacity values of composites reduce due to the increasing of the reinforcement addition. The maximum reduction of heat capacity is obtained in Ti6Al4V/10% Nano-SiCp composites.

4.3.3 Total Heat

It is observed that, the total heat of the samples increases with an increase in temperature. From Figures 4.29-4.36, it is observed that the total heat calculated for alloy and composites linearly increase with an increase in temperature. Further, the linear reduction of total heat is observed when the addition of percentage volume of reinforcement with the alloy increases. The total heat values reduce due to the increasing of the reinforcement addition. It is observed that in the second and third runs total heat are nearly same when compared with first run. This is because of removal of the process-induced residual stresses and the surface absorbed moisture in the specimen during the first run. [90, 132]

![Temperature Vs Total Heat](image.png)

*Fig. 4.29 Total heat of run effects of Ti6Al4V alloy (Sample-1)*
Fig. 4.30 Total heat of run effects of Ti6Al4V alloy (Sample-2)

Fig. 4.31 Total heat of run effects of Ti6Al4V//10% Nano-SiCp composites (Sample-1)
Fig. 4.32 Total heat of run effects of Ti6Al4V/10% Nano-SiCp composites (Sample-2)

Fig. 4.33 Total heat of run effects of Ti6Al4V/15% Nano-SiCp composites (Sample-1)
Fig. 4.34 Total heat of run effects of Ti6Al4V/15% Nano-SiCp composites  
(Sample-2)

Fig. 4.35 Total heat of run effects of Ti6Al4V/20% Nano-SiCp composites  
(Sample-1)
4.3.4 Effect of Addition of Different Volume Fraction of Nano-SiCp with Ti6Al4V Alloy in Total Heat Measurement

It is similar to the heat capacities of materials; the total heat measured also shows variations. The total heat of the Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy in the measured temperature range. The total heat shows a linear variation when the temperature increases. The size of the nano-SiCp has influence on the total heat of the composite materials [90, 132]. The Figures 4.37-4.42 shows the variations of the total heat of the Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites.
Fig. 4.37 Total heat of Ti6Al4V alloy & Ti6Al4V /Nano-SiCp composites  
(1st sample in 1st run)

Fig. 4.38 Total heat of Ti6Al4V alloy & Ti6Al4V /Nano-SiCp composites  
(2nd sample in 1st run)
Fig. 4.39 Total heat of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites
(1\textsuperscript{st} sample in 2\textsuperscript{nd} run)

Fig. 4.40 Total heat of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites
(2\textsuperscript{nd} sample in 2\textsuperscript{nd} run)
Fig. 4.41 Total heat of Ti6Al4V alloy & Ti6Al4V Nano-SiCp composites (1\textsuperscript{st} sample in 3\textsuperscript{rd} run)

Fig. 4.42 Total heat of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2\textsuperscript{nd} sample in 3\textsuperscript{rd} run)

From Figure 4.39-4.42 it is observed some contentious change in total heat of Ti6Al4V/15%Nano-SiCp composites and Ti6Al4V/20%Nano-SiCp composites in first
sample and second sample like heat capacity values. But in the case of Ti6Al4V/10%Nano-SiCp composites all the measured values trends are in similar patterns with unswerving values in both first and second samples. Even though the total heats of all the composites are less than the alloy in second run on-wards. The variations in total heats of alloy and all composites are due to the presence of the nano-SiCp in the composites. The presence of the nano-SiCp reduces the total heat. During the total heat measurement, alloy and composites shows, similar pattern of linear variations at same temperature values. The total heat values of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites are significantly varies under the identical conditions. Total heat of Ti6Al4V/Nano-SiCp composites are less than the Ti6Al4V alloy, which is due the fluctuations in the trend indicate the misfit strains introduced in the MMCs by the particulate reinforcement and reduction in the interface area compared to a uniform geometry[90,132] The maximum reduction of total heat is obtained in Ti6Al4V/10% Nano-SiCp composites.

4.4 STUDY OF COEFFICIENT OF THERMAL EXPANSION OF Ti6Al4V ALLOY AND Ti6Al4V/Nano-SiCp COMPOSITES USING THERMO MECHANICAL ANALYZER (TMA)

The metal-matrix composites (MMCs) has been developed indigenously and tested in Thermo Mechanical Analyser (TMA). This study revel the measurement method and thermo physical properties of MMCs.[97, 139]

The coefficient of thermal expansion (CTE) measurements were carried out on two samples of each materials, Ti6Al4V alloy and Ti6Al4V alloy with different volume percents of nano-SiCp (10, 15 and 20% of nano-SiCp) composites were used for investigation. CTE measurement of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites (TACTE1, TACTE2, 10CTE1, 10CTE2, 15CTE1, 15CTE2, 20CTE1 and 20CTE2) were carried out thrice in all eight samples.

Instead of pre-conditioning the samples in a separate furnace, the first run carried out inside the module which 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 and third run were better, repeatable, reproducible and reliable. The heating rate selected was 10°C/min from -100°C to 400°C. Nitrogen was used as a purge gas at the flow rate of 50 ml/min. A small static force (0.05 N)
was applied on the sample by the measuring probe. The TMA traces for the dimension change and coefficient of thermal expansion (CTE) as a function of the temperature were plotted.

4.4.1 Scanning Run Effect

The dimension change and coefficient of thermal expansion of Ti6Al4V Alloy and Ti6Al4V/Nano-SiCp composites were measured and shown in Figures 4.43-4.58. The dimension changes as a function of the temperature were drawn. The interaction between the matrix and reinforcement will also influence the resultant coefficients of thermal expansion and dimension change of the composites.

From the Figures 4.43-4.50, it is observed that for all Ti6Al4V Alloy and Ti6Al4V/Nano-SiCp the dimension change measured increases linearly with increase in temperature [97, 137]. In the 1st run, the measured dimension change shows some deviations which are due to the presence of process-induced residual stresses and the surface absorbed moisture in the samples. In the 2nd and 3rd runs, these effects are removed from the samples and the measured dimension changes are almost similar.

From the Figures 4.51-4.58, it is observed that for all Ti6Al4V Alloy and Ti6Al4V/Nano-SiCp Composites, the coefficient of thermal expansion measured increases with increase in temperature. But, the increase is not perfectly linear [97]. In the 1st run, due to the presence of process-induced residual stresses and the surface absorbed moisture in the samples, the measured coefficient of thermal expansion shows some deviations. In the 2nd and 3rd runs, these effects are removed from the samples and the measured coefficients of thermal expansion are almost similar.
Fig. 4.43 Dimension change of run effects of Ti6Al4V alloy (Sample-1)

Fig. 4.44 Dimension change of run effects of Ti6Al4V alloy (Sample-2)
Fig. 4.45 Dimension change of run effects of Ti6Al4V/10%Nano-SiCp composites, (Sample-1)

Fig. 4.46 Dimension change of run effects of Ti6Al4V/10%Nano-SiCp composites, (Sample-2)
Fig. 4.47 Dimension change of run effects of Ti6Al4V/15%Nano-SiCp composites, (Sample-1)

Fig. 4.48 Dimension change of run effects of Ti6Al4V/15%Nano-SiCp composites, (Sample-2)
Fig. 4.49 Dimension change of run effects of Ti6Al4V/20%Nano-SiCp composites, (Sample-1)

Fig. 4.50 Dimension change of run effects of Ti6Al4V/20%Nano-SiCp composites, (Sample-2)
Fig. 4.51 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V alloy (Sample-1)

Fig. 4.52 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V alloy (Sample-2)
Fig. 4.53 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/10%Nano-SiCp composite (Sample-1)

Fig. 4.54 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/10%Nano-SiCp composite (Sample-2)
Fig. 4.55 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/15%Nano-SiCp composite (Sample-1)

Fig. 4.56 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/15%Nano-SiCp composite (Sample-2)
Fig. 4.57 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/20%Nano-SiCp composite (Sample-1)

Fig. 4.58 Coefficient of thermal expansion (CTE) of run effects of Ti6Al4V/20%Nano-SiCp composite (Sample-2)
The comparison of the dimension change of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites with different volume fractions of nano-SiCp as a function of the temperature in 1st, 2nd and 3rd runs are shown in Figures 4.59-4.64.

4.4.2 Dimensional Change

From the Figures, 4.59-4.64 it is observed that for all Ti6Al4V Alloy and Ti6Al4V/Nano-SiCp Composites, the dimension change measured increases linearly with increase in temperature [137]. In the 1st run, the measured dimension change shows some deviations which are due to the presence of process-induced residual stresses and the surface absorbed moisture in the samples. In the 2nd and 3rd runs, these effects are removed from the samples and the measured dimension changes are almost similar. Most of the Ti6Al4V/Nano-SiCp composites show less dimension changes than the Ti6Al4V alloy in all the measured temperature range.

Fig. 4.59 Dimension change of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 1st run)
Fig. 4.60 Dimension change of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 1st run)

Fig. 4.61 Dimension change of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 2nd run)
Fig. 4.62 Dimension change of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 2nd run)

Fig. 4.63 Dimension change of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 3rd run)
The comparison of the coefficient of thermal expansion measurement of Ti6Al4V alloy and Ti6Al4V/ Nano-SiCp composites with different volume fractions nano-SiCp as a function of the temperature in 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> runs are shown in Figures 4.65-4.70. It is observed that for all Ti6Al4V Alloy and Ti6Al4V/ Nano-SiCp composites, the CTE measured increases linearly with increase in temperature [137]. In the 1<sup>st</sup> and 2<sup>nd</sup> set of samples, measured dimension change shows some deviations which was due to the presence of reinforcement in matrix alloy.

It is observed that the coefficient of expansion (CTE) of the samples increases with an increase in temperature. But, the increase is not perfectly linear and this can be attributed to an increase in the amplitude of vibration of the atoms of the constituents of the new material with increase in temperature. Due to the continuous programmed (controlled) heating, the MMCs attain the plastic state, where the activity due to residual stresses and change in void volume is predominant [138]. If the interface bonding is able to sustain the stresses, the overall CTE will be governed by the competition between the high CTE of the matrix material and the low CTE of the reinforcement particulates. It is inferred that CTE of the MMCs will be lower than the matrix material due to the reinforcement. MMCs will have a
range of CTE values depending on the volume fraction and the bonding strength of the reinforcement interface.

When the temperature is changed, both the matrix material and the reinforcement are strained elastically and the plastic flow is needed to accommodate the increase in the volume of the MMCs specimen [138]. As a result, the stresses are built up at the matrix/reinforcement interface and the MMCs value reflects the overall bonding strength of the MMCs. Any degradation in the interface should contribute to a increase in the final CTE data measured.

Fig. 4.65 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites
(1st sample in 1st run)
Fig. 4.66 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 1st run)

Fig. 4.67 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample in 2nd run)
Fig. 4.68 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2\textsuperscript{nd} sample in 2\textsuperscript{nd} run)

Fig. 4.69 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1\textsuperscript{st} sample in 3\textsuperscript{rd} run)
Fig. 4.70 CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample in 3rd run)

Fig. 4.71 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample 1st run)
Fig. 4.72 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample 1st run)

Fig. 4.73 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1st sample 2nd run)
Fig. 4.74 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2\textsuperscript{nd} sample 2\textsuperscript{nd} run)

Fig. 4.75 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (1\textsuperscript{st} sample 3\textsuperscript{rd} run)
Fig. 4.76 Comparison of CTE of Ti6Al4V alloy & Ti6Al4V/Nano-SiCp composites (2nd sample 3rd run)

It is observed that, the increase in the volume % of the SiC particulates brings down the dimension change value of the MMCs at the measured range thereby the coefficient of thermal expansion of Ti6Al4V/Nano-SiCp composites also reduced, which is shown in Figure 4.71 – 4.76, it is inferred that CTE values of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites are significantly varies under the identical conditions. The CTE of the composites are less when compared to alloy for any given measured temperature range due to the interface bonding is able to sustain the stresses, the overall CTE will be governed by the competition between the high CTE of the matrix material and the low CTE of the reinforcement particulates.[138,139]. Denoath et al.[141] also obtained experimental results indicating that an increase in the content of ceramic particulates reduces the CTE of a MMC. And also these observations have been confirmed by Dellis et al. [142] who obtained similar results in their work.

4.4.3 Effect of Addition of Different Volume Fraction of Nano-SiCp with Ti6Al4V Alloy

A decrease in particle size (i.e., more surface area for oxides to form) also resulted in reduced CTE. Xu et al. [144] studied the CTE behavior of Al/TiCp composites made by the XDTM process. They define an interfacial zone (not a reaction zone) which provided a significant constraint on the CTE. Using a three-component rule of mixtures model, particle,
matrix, and interface layer, the experimental CTE was predicted quite well. The effect of particle size was captured by this model, because the interfacial layer increased with decreasing particle size (increasing interfacial area).

Chawla et al [116] explained that the CTE of particle reinforced MMCs is affected by a variety of factors, such as interfacial reactions, plasticity due to CTE mismatch between particle and matrix, during heating or cooling, and residual stress.

Recently, it has been demonstrated that the CTEs of alloys and composites are mainly changed by a volume fraction and shape of reinforcements [112, 129]. The CTE of the composite is generally less than the rule of mixtures value because the presence of ceramic particles (usually of low CTE) introduces a constraint on the expansion of the metallic matrix (usually of high CTE). In this regard, it is important to point out that interface can exert some influence on the value of CTE, especially for very small particle size. The CTE is sensitive to particle distribution (clustering). In addition to the interface, the CTE of particle reinforced MMCs is affected by several other factors. These include: plasticity due to CTE mismatch between reinforcement and matrix, during heating or cooling; reinforcement fracture, residual stress, and local stresses at points of contact between reinforcement [4].

Lee et al [140] postulated that an incompressible plastic layer may form at the particle/matrix interface and result in lower expansion of the matrix and thus, the overall composite. The CTE may also be affected by the particle fracture. An increase in particle fracture resulted in increased CTE. Residual stress also has an important effect on CTE.

The variation in CTE with the increase in the volume percentage of the reinforcements in the MMC falls drastically. The rise in CTE value above 70 °C is less mainly due to the plasticization effects in the MMCs. The high volume fraction MMCs become strained elastically by expanding or contracting the Titanium matrix, which depends on the energy needed for the increase in plastic flow once the change is exceeded.

In general, the CTE of a composite is different from that given by a simple rule of mixtures \((\alpha_1 V_1 + \alpha_2 V_2)\). This is because the presence of reinforcement, with an expansion coefficient less than that of matrix, introduces a mechanical constraint on the matrix. The degree of constraint is also dependent on the nature of the reinforcement, e.g., a fiber will cause a greater constraint on the matrix than a particle[4].

Denoath et al [141] also obtained experimental results indicating that an increase in the content of ceramic particulates slightly reduces the CTE of a MMC. And also these
observations have been confirmed by Dellsis et al. [142] who obtained similar results in their work.

It is worth noting that the numerical calculations reported by Shen. Y.L et al. [130] are fully consistent with the present experimental results. This observation is also in complete agreement with the results of Arpon. R et al. [143], which indicate that the only relevant parameter is the particle volume fraction (no matter whether the composite has a single particle size or a bimodal size distribution).

The great difference between the coefficients of thermal expansion of the titanium and the SiC particles induces stresses and increases the dislocation density in the metal. As a result, the metal is hardened up to an extent and is greater in the higher the reinforcement content.

Xu et al. [144] were of the opinion that the lattice distortion at the interface affects the CTE value of the composite. Since the interfacial area is a function of the reinforcements shape and size, the CTE of the composite will vary with the reinforcement shape and size.

Strengthening through changes in the matrix microstructure as a result of the addition of particles has been reported to occur through the combination of several mechanisms including coefficient of thermal expansion (CTE) mismatch, grain and sub grain refinement, increased work hardening and change in precipitate strengthening [145-148].

Mahagundappa M. Benal and H. R. Shivanand [107] concluded that the residual thermal stresses at the matrix reinforcement interface are induced by the CTE mismatch between the reinforcement and matrix, which are dependent on the properties of the matrix and reinforcements.

The increased dislocation density in the composite provides additional precipitate nucleation sites, thus accelerating the aging process [149]. Therefore, the increased number of barriers impeding dislocation motion, through increased grain and sub grain boundaries, dislocation density and precipitates and the presence of nano-SiCp are also argued to provide a significant contribution to strengthening [146, 147]. Both of these strengthening effects are amplified with increased volume fraction and decreased particle size which provide greater matrix-particle boundary area and decreased particle spacing [150].

A lattice distortion at the interface will affect the CTE value of the composites. Since the interfacial area is related to the particle size, the volume of such a lattice distortion layer
will depend on the particle size and accordingly, the effect of the particle size on the CTE of the composites will vary with the particle size as well as shape. For a given particle volume fraction, the smaller particle size, the greater the volume fraction of interfacial zone. The effect of particle size on the interfacial zone size for a given volume fraction of particle were also reported by Xu et al. [144].

This interfacial zone will be constrained to expand or contract with the particle, and thus, CTE of this zone will be closer to that of the particle than that of the metal matrix. In order to explain the effect of the particle size on the CTE of the particle reinforced metal matrix composite, consider that the composite consists of a matrix phase, a particulate reinforcement and an interfacial zone to which a CTE different from that of the matrix can be ascribed. In this approximation, the constraint on the matrix expansion is thus embedded in the interfacial zone. It can be writing for the CTE of a three-component composite as follows:

\[
\alpha_i = V_p\alpha_p + V_m\alpha_m + V_i\alpha_i
\]

(4.2)

where the subscripts, c, p, m, and i, denote the composite, particle, matrix, and interfacial zone, respectively. The interfacial zone volume fraction, \(V_i\), was calculated by the interfacial area and the thickness of the lattice distortion layer.

Elomari et al. [151] studied the CTE behavior of SiC particle reinforced pure Al matrix composites and concluded that decrease in particles size (i.e., more surface area for oxides to form) resulted in reduced CTE.

In addition to the interface, the CTE of particle reinforced MMCs is affected by several other factors. These include: plasticity due to CTE mismatch between reinforcement and matrix, during heating or cooling; reinforcement fracture, residual stress, and local stresses at points of contact between reinforcement.

An incompressible plastic layer may form at the particle/matrix interface and result in lower expansion of the matrix and thus, the overall composite. For a good heat material, it is desirable to have low CTE and \(C_p\) property [137].
4.5 MODELLING AND SIMULATION OF THERMAL PROPERTIES OF Ti6Al4V ALLOY AND Ti6Al4V/Nano-SiCp COMPOSITES

Thermal analysis of composites is always an active issue in composite mechanics. The simulations were carried out by using the finite element program ANSYS. The thermal properties and physical properties of individual constituents could be used as input parameters in the finite element model to calculate the effective properties of composites. The composites were simulated by a three-dimensional model.[152-154]

4.5.1 Finite Element Simulation on Specific Heat Capacity

Thermal calculation using finite element method is employed to solve the temperature dependent specific heat capacity via ANSYS and the three dimensional transient heat transfer problem was solved. In this study, the specific heat capacity of the Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites in the temperature range of 173K to 648K are determined.[158, 159]

Cylinder was taken for the nodal solution of the specimen with minimum result value on plot (SMN) as 173K and maximum value on the plot (SMX) as 648K. The specific heat value predictions compared with each other, with the available experimental data and with the finite element results of Ti6Al4V alloy and Ti6Al4V / Nano-SiCp composites and are shown in Figures 4.77 – 4.80.
Fig 4.77. FEM model of Ti6Al4V alloy specific heat capacity

Fig 4.78. FEM model of Ti6Al4V/10% Nano-SiCp specific heat capacity
Fig. 4.79 FEM model of Ti6Al4V/15% Nano-SiCp specific heat capacity

Fig. 4.80 FEM model of Ti6Al4V/20% Nano-SiCp specific heat capacity
Heat capacity predictions compared with the available experimental data and with the finite element results of Ti6Al4V alloy and Ti6Al4V / Nano-SiCp composites. The comparisons of these model predictions with experimental data hold good agreement with each other and minor discrepancies are also notified. The heat capacity predictions using FEM is shown in Table. [4.1]

<table>
<thead>
<tr>
<th>Material</th>
<th>Ti6Al4V Alloy</th>
<th>Ti6Al4V/10% Nano-SiCp</th>
<th>Ti6Al4V/15% Nano-SiCp</th>
<th>Ti6Al4V/20% Nano-SiCp</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deflection</td>
<td>0.010626</td>
<td>0.009228</td>
<td>0.009246</td>
<td>0.010082</td>
</tr>
</tbody>
</table>

From the Tables 3.8 – 3.15 and Table 4.1, it is observed that the Cp values of alloy and composites increases with increase of reinforcement particles as observed from the experimental values. The experimental heat capacity value of Ti6Al4V/10%Nano-SiCp Composite has good agreement with model prediction data. The Cp values of Ti6Al4V/10%Nano-SiCp composite is low when compare with alloy and other composites. For a good heat material, it is desirable to have low Cp property [90,137].

4.5.2 Finite Element Simulation on Coefficient of Thermal Expansion

Linear thermal expansion is the fractional change in length of a body when heated or cooled through a given temperature range and usually it is given as a coefficient per unit temperature interval, either as an average over a stated range, or as the tangent to the expansion curve at a given temperature. The longitudinal and transverse coefficients of thermal expansion of the orthotropic unidirectional composites must be known for design purposes. These composite properties can be experimentally measured, which can be expensive and time consuming when evaluating many different material systems, or predicted using the thermal and mechanical properties of the constituents. Furthermore, as a result of the increasing computer technology, numerical solutions such as finite element analysis are being used to determine the thermal expansion coefficients of composite materials. [155, 160]
Fig.4.81. FEM model of Ti6Al4V alloy CTE

Fig.4.82. FEM model of Ti6Al4V/10% Nano-SiCp CTE
Cylinder was taken for the nodal solution of the specimen with minimum result value on plot (SMN) as 173K and maximum value on the plot (SMX) as 673K. The CTE predictions compared with each other, with the available experimental data and with the finite element results of Ti6Al4V alloy and Ti6Al4V/ Nano-SiCp composites and are shown in Figures 4.81 – 4.84

CTE predictions compared with the available experimental data and with the finite element results of Ti6Al4V alloy and Ti6Al4V / Nano-SiCp composites. The comparisons of these model predictions with experimental data hold good agreement with each other and minor discrepancies are also notified. The CTE predictions using FEM is shown in Table. [4.2]

Table 4.2. Model prediction deflection values (CTE)

<table>
<thead>
<tr>
<th>Material</th>
<th>Ti6Al4V Alloy</th>
<th>Ti6Al4V/10% Nano-SiCp</th>
<th>Ti6Al4V/15% Nano-SiCp</th>
<th>Ti6Al4V/20% Nano-SiCp</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deflection</td>
<td>0.023164</td>
<td>0.019585</td>
<td>0.020072</td>
<td>0.020895</td>
</tr>
</tbody>
</table>

From the Tables 3.16 – 3.23 and Table 4.2, it is observed that the CTE values of alloy and composites increases with increase of reinforcement particles as observed from the experimental values. The experimental CTE value of Ti6Al4V/10\% Nano-SiCp composite has good agreement with model prediction data. The CTE values of Ti6Al4V/ 10\% Nano-SiCp composite is low when compared with alloy and other composites. An incompressible plastic layer may form at the particle/matrix interface and result in lower expansion of the matrix and thus, the overall composite. For a good heat material, it is desirable to have low CTE property [97,137].

Summary:

Nano silicon carbide powder was prepared by high energy ball mill. Ti6Al4V Alloy with different volume fractions (0, 10, 15 and 20\%) of nano-SiCp reinforced metal matrix composite materials were fabricated through powder metallurgy technique. Thermal analysis of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp composites were conducted to explore the stability and behavior of these materials for property development. Effect of addition of nano-SiCp with Ti6Al4V alloy is reported. The favorable and instable temperature range of materials have been characterized and analyzed. Also a FE model has been developed by ANSYS to analyze the temperature distribution and deflection of Ti6Al4V alloy and Ti6Al4V/Nano-SiCp Composites. Thermal property predictions are compared and validated.
The 6000 series Aluminium alloy has been used for aircraft applications. The average CTE of aluminium alloy is 25.2 μm/(m°C)[161]. The average CTE of Ti6Al4V alloy is 9.16 μm/(m°C) [3]. So, the target CTE values of Ti6Al4V/Nano-SiCp composites are must be less than 9.16 μm/(m°C). But, in this study, the new fabricated Ti6Al4V/Nano-SiCp composites has the average CTE is found to be 7.876 μm/(m°C). Hence Ti6Al4V/Nano-SiCp composites may be used instead of Ti6Al4V alloy and Aluminium alloy for the spacecraft applications.