Microstructural and Mechanical Characteristics of in-situ Titanium Metal Matrix Composites

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Abstract – Discontinuously reinforced titanium matrix composites (DRTMCs) are emerging as an alternate to titanium and its alloys for ambient and elevated temperature applications. The present investigation deals with P/M processing of DRTMCs and their characterizat

I. INTRODUCTION

Discontinuously reinforced titanium matrix composites (DRTMCs) are of considerable interest for structural, thermal and armour applications due to the potential for making composites with high specific modulus and strength at relatively low costs. Ranganath et al. [1] produced Ti-TiB-TiC composites by combustion assisted synthesis of Ti-B₄C (CAS) using arc melting technique. This process could not be followed for synthesizing composites having high volume fractions of reinforcements due to non uniformity in microstructures. Panda et al. and Sahay et al. [2,3] studied a series of TMCs, with varying volume fractions (30% to 90%) of TiB needles, made by hot pressing the Ti-TiB₂ powders. It was reported that morphology of TiB needle changes depending upon the reinforcement volume fraction in the composite. Bhat et al. [4] produced in-situ TMCs containing high volume fractions of TiB, TiB-TiC, by hot pressing the Ti-TiB₂ and Ti-B₂C powders, respectively. Their study reported formation of TiB needles in former case while TiB particles in the latter case. Ma et al. [5] prepared Ti-TiB composites with low volume fractions (<15%) of reinforcements by reaction hot pressing the Ti-B, Ti-TiB₂, Ti-B₂C and Ti-BN. The hot pressing route followed by the above researchers is expensive and has size & shape limitations. The present investigation is aimed at evaluation of pressureless sintering process to synthesize Ti-TiB-TiC composites out of Ti-B₂C cold pressed compacts and to study the effect of vol.% of the reinforcement on the microstructural features as well as on the mechanical properties.

II. EXPERIMENTAL PROCEDURE

Ti-TiB-TiC composites have been synthesized by pressureless reaction sintering of Ti-B₂C green compacts making use of the following reaction.

5 Ti + B₄C → 4 TiB + TiC \( \Delta H_{298} = -754.36 \text{ kJ/mol} \) … (1)

Excess titanium over and above the stoichiometric titanium content determined by Eq (1) was used to get necessary amount of matrix titanium. Powders of titanium and three different vol.% of B₄C (1.8, 3.6 and 5.4) have been used to synthesize 10, 20 and 30 vol.% (TiB + TiC) reinforcements, respectively in titanium matrix.

Shrinkage of 16-18% has been observed in the...
dimensions of the cold compact as a result of sintering. Phillips PW 3020 diffractometer with Cu Kα radiation was used to conduct X-ray diffraction studies on pressureless sintered compacts of titanium and composites using for confirmation of phases before and after sintering process. LEO 440i Scanning Electron Microscope (SEM) has been used to study the morphology and distribution of the reinforcements in the un-etched as well as etched composite samples. Flexural strength of the composite and un-reinforced titanium specimens in three point bending mode has been determined. Vickers hardness of the composite and un-reinforced titanium specimens using 5 kg load has been measured. The elastic moduli of the composites and unreinforced titanium specimens of 4 mm x 4 mm x 50 mm was studied by using dynamic methods based on impulse excitation of vibrations (Buzz Mac. Intl. USA).

III. RESULTS & DISCUSSION

3.1 XRD & Microstructure

X-ray diffractogram of the pressureless sintered composite and titanium samples is illustrated in Fig. 1. Presence of Ti, TiB, TiC peaks and absence of B,C peak in Fig. 1 confirms completion of the reaction between Ti-B,C during sintering. Highly exothermic reaction (ΔH_form = -754.36 kJ/mol) between the reactants helps in completion of the reaction [6]. Fig. 2(a) shows the SEM image of sintered titanium. Fig. 2(b) (10 vol.% TiB+TiC) shows the presence of primary and secondary needles of TiB distributed uniformly. The population density of the TiB needles increased as seen in Fig. 2(c) (20 vol.% TiB+TiC) with some amount of refinement in the structure. Fig. 2(d) (30 vol.% TiB+TiC) illustrates the presence of needle shaped randomly oriented TiB whiskers in the titanium matrix and colonies of densely packed short TiB whiskers. The short TiB whisker colonies appear to be interconnected and uniformly distributed in the matrix. The needle shaped morphology of TiB whiskers observed in the microstructure has been explained in terms of crystal structure and boron diffusion mechanism for TiB [2]. Sahay et al. [3] categorized such TiB needles with different sizes as primary and secondary needles and proposed a mechanism for formation of TiB in different aspect ratios and diameters in terms of preferential growth rate of TiB along [010] direction due to higher density of strong B-B bonds and one way diffusion of B along this direction. Sahay et al. proposed a critical volume fraction of TiB₂ (29 vol.%) above which thin secondary needle shaped TiB starts growing. However, secondary TiB needles were noticed even at 10 vol.% of (TiB + TiC) reinforcements in the present study. Use of finer (13 µm) titanium powder and 2.4 µm TiB₂ powder (size ratio of 11.6:1) used by Sahay et al [3] has resulted in localized increase in concentration of boron. In addition longer duration required for completion of the reaction between titanium and coarser B₄C resulted in smaller mean free path for the growth of primary needles, promoted the growth of secondary TiB needles.

Figure 1 : X-Ray diffraction patterns for both pressureless sintered titanium and composite samples having different vol.% of (TiB + TiC).
3.2 Mechanical Properties

Table 1 shows the mechanical properties of the composites and unreinforced titanium samples processed under similar conditions. Sintered sample of unreinforced titanium exhibited average flexural strength of 234 MPa. With increase in reinforcement content the flexural strength of the composites are increasing. It is worth noting that the flexural strength of the composite with 30 vol.% reinforcement is almost double the strength of the unreinforced titanium. Marginally lower flexural strength obtained for Ti-30 vol.% (TiB+TiC) composite (614 MPa) when compared to the flexural strength of hot pressed Ti-70% (TiB+TiC) composite (629 MPa) reported elsewhere [4], indicates that pressureless sintering process is potential alternative method to VHP method for synthesizing Titanium Matrix Composites. Similar trend can also be observed in case of hardness of the composites. Presence of TiB in the form of long single crystalline whiskers or needles with clean interface is responsible for the improved flexural strength and hardness according to the theories of whisker induced stiffening and strengthening [7].

<table>
<thead>
<tr>
<th>Material</th>
<th>Flexural Strength (MPa)</th>
<th>Vickers Hardness (VHN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium</td>
<td>234</td>
<td>415</td>
</tr>
<tr>
<td>Ti-10% (TiB+TiC)</td>
<td>447</td>
<td>526</td>
</tr>
<tr>
<td>Ti-20% (TiB+TiC)</td>
<td>578</td>
<td>572</td>
</tr>
<tr>
<td>Ti-30% (TiB+TiC)</td>
<td>614</td>
<td>690</td>
</tr>
</tbody>
</table>

Elastic modulus measured for the composites by pulse excitation technique are reported in Table 2. Average elastic modulus of 131 GPa exhibited by the sintered samples of unreinforced titanium in the present study, is in between the elastic modulus values of 100 – 145 GPa reported for the transverse and longitudinal directions of hexagonal close packed unit cell of high purity titanium [8].

There is a gradual increase in the elastic modulus values from 155 GPa to 188 GPa for composites with increasing reinforcement content from 10 to 30 volume%. Elastic modulus values obtained in the present study have been compared with those obtained using different models as shown in the Fig. 3. While calculating the elastic modulus values using various models, elastic modulus of TiB has been assumed to be 482 GPa [9]. It has been observed that present values are in between the values obtained by upper bound iso-strain model and lower bound iso-stress model. The observed values are matching with the values calculated by Tsai-Halpin equation by assuming the aspect ratio of (l/d) = 1. Whereas with the observed average aspect ratio value of (l/d) = 10, the calculated elastic modulus values are deviating from the observed values. This indicates that even though most of the reinforcement
needles have l/d ratio greater than critical l/d ratio defined for effective load transfer, isotropic distribution of the needles and their clusters are responsible for obtaining lower Young’s modulus values than the predicted values by the model. Moreover Tsai-Halpin model is more valid for composites having uniformly distributed reinforcements. Similar observations were reported by Ranganath et al. [10], where variation in Young’s modulus with volume fraction of TiB + Ti3C reinforcements in titanium matrix has been studied. Using the rule of mixtures for continuous reinforcements, a large difference in the calculated modulus values was observed for iso-strain and iso-stress models [10]. By using Tsai - Halpin equation for discontinuous reinforcements it was shown that the equation is valid for only particle aspect ratios nearing unity. However, with particles having high aspect ratios (l/d) of 10 – 20 the equation seems to be invalid, which is in good agreement with the results obtained in the present investigation.

Table 2 : Elastic modulus values for composites

<table>
<thead>
<tr>
<th>Vol.%</th>
<th>Elastic Modulus (GPa)</th>
<th>Iso-Strain</th>
<th>Iso-Stress</th>
<th>Tsai-Halpin (l/d)=1</th>
<th>Tsai-Halpin (l/d)=10</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>155</td>
<td>166</td>
<td>141</td>
<td>150</td>
<td>162</td>
</tr>
<tr>
<td>20</td>
<td>172</td>
<td>201</td>
<td>153</td>
<td>172</td>
<td>195</td>
</tr>
<tr>
<td>30</td>
<td>188</td>
<td>236</td>
<td>167</td>
<td>196</td>
<td>228</td>
</tr>
</tbody>
</table>

Figure 3 : The relation between reinforcement volume fraction to the Elastic Modulus of the composites.

IV. CONCLUSIONS

The following conclusions emerge from the present work:

1. Ti – TiB – TiC composites are processed using titanium and B4C powders as raw materials by pressureless sintering technique.
2. XRD studies confirmed the presence of TiB and TiC reinforcements and absence of B4C in titanium matrix indicating the completion of the in-situ reaction between titanium and B4C.
3. Microstructure of sintered Ti – (TiB + TiC) composites consists of primary and secondary needles of TiB in the form of isolated needles, and equiaxed TiC particles.
4. Sintered Ti – TiB – TiC composites exhibited improved mechanical properties over sintered titanium compacts processed under similar conditions.

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VI. REFERENCES


