Development of Ti-TiC composites by powder metallurgy route using recycled machined Ti chips

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Abstract
A large amount of waste in the form of chips is generated during production processes like machining, grinding or cutting of Ti/Ti based alloy. From the economic, strategic and environmental point of view, these chips should be reused and recycled. Powder metallurgy provides a very suitable and economical way for the utilization of these chips. Initially, Ti chips were cleaned with acetone, cut into small pieces and then milled in a high energy planetary mill. Milling was carried out using stainless steel ball in a stainless steel jar. Wet milling was conducted where chips and balls were immersed under toluene to prevent oxidation. Milling was carried out for two hours and then Ti powders were characterized by XRD, SEM and TEM. The prepared Ti powder and graphite powder were mixed in different weight percentage (48:12, 100:6) and then milled in planetary mill. The milled powders were compacted at different pressure (400-700 MPa) by using a powder compaction press and then sintered at different temperatures (1000-1200 °C) in a tubular furnace under argon gas. It has been found that the weight ratio of Ti and C (graphite) has great influence on the formation of TiC in the produced composite. The phase study and microstructural characterization of Ti-TiC composites were characterized by XRD and SEM. Experimental results show that with the increase in compaction pressure and sintering temperature; the density, micro-hardness and wear resistance of the Ti-TiC composite increases.

Keywords: Ti scraps, Ti-TiC, metal matrix composite, planetary milling, powder metallurgy
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Introduction

- Most production processes of Titanium tools like machining and forging generate large amount of scraps in the form of chips. From the economic, strategic and environmental point of view, these chips should be reused and recycled.
- Powder metallurgy provides a very suitable and economical way for the utilisation of these chips.
- This research work will investigate the production of Titanium powder from the waste chips of the same and the consolidation of that powder.
- The processes involved are planetary milling of chips, spark plasma sintering, microstructure and mechanical property evaluation.
Experimental procedure

Cleaning of Ti chips with acetone

Planetary milling of Ti chips in planetary mill (DDPM): wet milling (toluene), BPR: 10:1, Steel ball dia.: 8 mm, Time-2.5 h

Characterization of Ti milled powder: XRD, FESEM and TEM

Milling of Ti and graphite powder in planetary mill: 12 h

Characterization of Ti + graphite milled powder: XRD, FESEM and TEM

Consolidation of powder by spark plasma sintering and characterization
Results & Discussion: Synthesis of Ti powder

XRD spectra of Ti chips after planetary milling

- One weak peak of Fe is visible with all Ti peaks
- There is broadening of peaks

- Particles are flattened after 0.5 h of milling but refinement of powder takes place with progress of milling

SEM micrographs of Ti chips milled for (a) 0.5 h (b) 1 h (c) 1.5 h and (d) 2.5 h
‘d’ spacing = 0.28 nm = 2.80 Å (Hexagonal Ti)
Standard “d” value of Hexagonal Ti for (001) is 2.8190 Å (JCPDS File no: 51-0631)
Planetary milling of Ti+ graphite powder

XRD spectra of Ti+ graphite powder after milling
FESEM micrographs after SPS

- Higher amount of spherical pores are present in sample sintered at 900 °C
- There is contamination of Fe and Cr, as milling was carried out using steel balls and stainless steel jars

<table>
<thead>
<tr>
<th>El AN Series</th>
<th>Weight % [wt.%]</th>
<th>Atom. % [at.%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti 22 K-series</td>
<td>56.03</td>
<td>41.43</td>
</tr>
<tr>
<td>C 6 K-series</td>
<td>13.07</td>
<td>38.52</td>
</tr>
<tr>
<td>Fe 26 K-series</td>
<td>19.93</td>
<td>12.63</td>
</tr>
<tr>
<td>Cr 24 K-series</td>
<td>9.41</td>
<td>6.41</td>
</tr>
<tr>
<td>Mn 25 K-series</td>
<td>1.57</td>
<td>1.01</td>
</tr>
</tbody>
</table>

Total: 100.00 100.00
Conclusions

• Ti powder can be generated by planetary milling of Ti chips for 2.5 h
• Powder particles of size 10-20 µm are obtained after milling
• There is iron contamination of 2.37 wt. % after 2.5 h of milling
• There is formation of TiC after milling Ti and graphite powder for 12 h. Iron contamination increases with milling.
• Samples sintered by SPS at 900 °C exhibits higher amount of pores than sintered at 1000 °C.
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