### Synthesis of Ti -TiC nano composite from waste Ti machining chips by planetary milling followed by spark plasma sintering

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#### Abstract

Titanium is one of the fastest growing materials used in aerospace applications due to light weight. Most production processes like machining and forging generate large amount of titanium and titanium alloy scrap in the form of chips. From the economic, strategic and environmental point of view, these scrap should be reused and recycled. Powder metallurgy provides a very suitable and economical way for the utilization of waste titanium scraps. In the present investigation, an attempt has been made to prepare Ti-TiC nano composite from Ti chips and graphite powder by planetary milling. The Ti-TiC nano composite has also been consolidated by spark plasma sintering.

Planetary milling is carried out in a high energy dual-drive planetary mill (DDPM) using stainless steel jar of 1 L volume and stainless steel ball of 10 mm diameter. Jar volume of 30 % is filled with cleaned Ti waste chips, graphite powder and steel ball. Ti chips and graphite powder are taken in 50:50 atomic ratio and ball to powder/chips weight ratio of 10:1 is maintained. Milling is carried out in dry condition and argon gas is purged before milling. Milling is carried out for a total period of 3 hours at the intervals of 30 minutes. At each interval, argon gas is purged without opening steel jar. Powder is characterized by X-Ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The powders obtained are irregular in shape with a crystallite size of 5-10 nm. Islands of TiC and Ti<sub>2</sub>C are observed in a matrix of Ti.

Finally, 3 hours milled powder is sintered by spark plasma sintering (SPS). SPS is carried out at 900 and 1000°C for 5 minutes at a pressure of 50 MPa. It has been found that sample sintered at 1000°C exhibits higher density and hardness than sintered at 900°C. There is a considerable increase in hardness from 378 to 614 HV on increasing the temperature from 900 to 1000°C.

Keywords: Ti-TiC nano composite, planetary milling, spark plasma sintering





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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 utilization 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.





## Motivation

- Recycling of waste Ti machining chips
- Conversion of Ti chips into Ti powder for powder metallurgy applications
- Fabrication of Ti-TiC nano composite from waste Ti chips
- Characterization of Ti-TiC nano composite powder mixture
- Consolidation of Ti-TiC nano composite by spark plasma sintering (SPS)
- Microstructural characterization and mechanical property evaluation of Ti-TiC nano composite

# **Experimental procedure**





### **Dual drive planetary mill**



**Stainless steel jar** 



**Ti chips** 

### Synthesis of Ti powder from waste Ti chips





As received Ti chips

### X-ray diffraction spectra of as received chips



#### Particle size distribution of Ti powder

### **Effect of Ball to Powder Weight Ratio**



### Morphology of powders: FESEM micrographs



**BPR= 10:1**; Average size of the particles: 27.5 μm **BPR= 20:1**; Average size of the particles: 13.4 μm

**Reduction in the particle size observed on increasing the BPR due to higher impact energy imparted by the balls.** 

## TEM micrographs









- Ti particles are submicron and irregular in shape
- "d spacing" matches with Ti
- SADP is ring pattern suggests particles are fine





•Both SADP confirm HCP Ti

• There is also contamination of Fe



#### SADP with BPR= 20:1



Spectrum: 1 spectrm raw.xls					
El AN Serie	s unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1	Sigma) [wt.%]
Ti 22 K-seri Cu 29 K-seri	es 100.00 es 0.00	100.00	100.00 0.00		3.03 0.00

3.03 0.00 \_\_\_\_

Total: 100.00 100.00 100.00





# Synthesis of Ti-TiC nano composite



XRD pattern of 3 hours milled Ti chips and graphite powder

# Morphology of Ti-TiC composite powder: FESEM micrographs



- Particles are almost spherical in shape
- Particle size is < 100 nm
- There is formation of agglomeration of fine powder particles

## Elemental mapping and EDS spectra







# **TEM micrographs**



### **Bright field TEM micrograph**

Dark field TEM micrograph

- Formation of nanoparticles (< 100 nm) after 2 hours of milling
- Particles are irregular in shape

### **Elemental mapping**







Γi-KA

Map data 1741

HAADF MAG: 105kx HV: 300kV





### EDS Spectra and SADP



- EDS spectra shows minor contamination of iron in Ti-TiC nano composite
- SADP exhibits the co-existance of Ti and TiC

### **Consolidation of Ti-TiC nano composite powder by SPS**



Compressive stress-strain curve 17

0.6

0.8

1.0

12

900°C 1000°C

# Conclusions

- Ti powder can be prepared after 3 hours planetary milling of waste Ti chips.
- Milling at BPR ratio of 20:1 generates finer powder than milling conducted at 10:1
- Ti-TiC nano composite is prepared by milling Ti chips and graphite powder after 2 hours of milling.
- Powder particles are nano meter size (<100 nm) and almost spherical in shape.
- Ti-TiC is fabricated by consolidation through spark plasma sintering (SPS).

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