Ultra-fine Grained and Dispersion-strengthened Titanium Materials Manufactured by Spark Plasma Sintering

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Abstract

Ultra-fine grained and dispersion-strengthened titanium materials (Ti-Si, Ti-C, Ti-Si-C) have been produced by high energy ball milling and spark plasma sintering (SPS). Silicon or/and carbon were milled together with the titanium powder to form nanometer-sized and homogeneously distributed titanium silicides or/and carbides as dispersoids, that should prevent grain coarsening during the SPS compaction and contribute to strengthening of the material. The microstructures and the mechanical properties showed that strength, hardness and wear resistance of the sintered materials have been significantly improved by the mechanisms of grain refinement and dispersion strengthening. The use of an organic fluid as carrier of the dispersoid forming elements caused a significant increase in ductility.

Keywords : dispersion strengthened titanium, mechanical properties, bimodal grain size distribution, wear behavior

1. Introduction

Titanium is well suited as metallic implant material, due to its best biocompatibility in comparison to other metals [1,2]. Disadvantages of using c.p.Ti as implant material are its low strength and insufficient hardness. Until now, the improvements of strength and wear resistance of titanium have been realized by alloying and by coating.

The aim of the present investigations was to obtain a significant increase in strength and hardness of titanium materials by developing a novel ultra-fine grained and dispersion-strengthened titanium material. The dispersoids are used to reduce the grain growth in the subsequent technologically necessary thermal treatment as well as to increase hardness and strength of the nanocrystalline titanium material. Titanium silicides and carbides are chosen as dispersoids because they have no deteriorating effect on the favourable TiO_2 passive layer and will not affect the body tissue.

2. Experimental and Results

As shown in previous investigations of the author group the granules with the desired nanocrystalline microstructure and finest dispersoids had been created by high energy ball milling of Ti-Si or Ti-C (graphite) powder mixtures in a RETSCH PM400 planetary ball mill [3].

With the aim of getting finer and more homogeneously distributed dispersoids **h**examethyldi**s**ilane – HS, $(CH_3)_6Si_2$, was used in the milling process [3] as carrier of the dispersiod forming elements Si and C. Prior to compaction, a heat treatment was necessary to remove the organic fluid

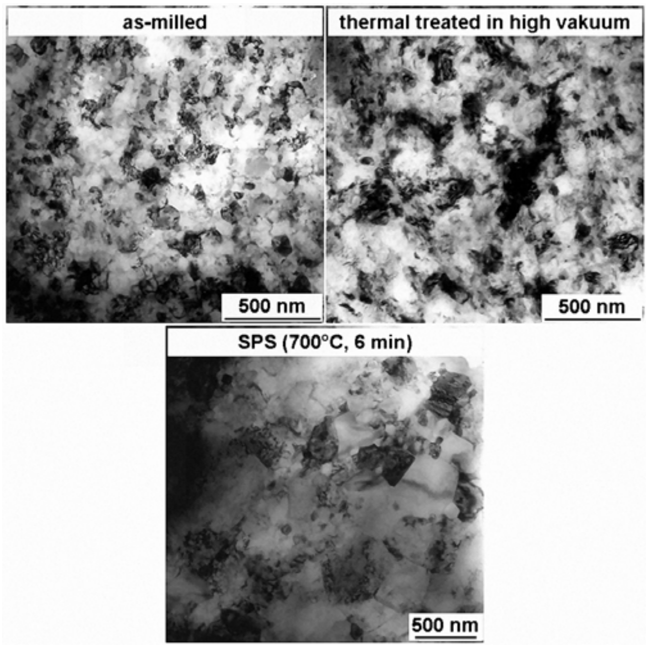

Fig. 1. TEM micrographs of Ti/HS after milling, high vacuum thermal treatment and SPS.

introduced hydrogen from the granules. Spark Plasma Sintering (SPS) was used to consolidate the powder granules. The microstucture of the material after the processing steps can be seen in Fig. 1.

Composition, processing parameters and bulk composition are shown in Table 1. The mechanical properties of the testet materials show a high strength and hardness caused by the effects of the Hall-Petch-relation and the Orowan mechanism. The poor ductility for Ti/1Si and Ti/1.5C created by powder mixtures could significantely be impoved by using HS for the milling process. For Ti/HS a plastic elongation of 9,6 % was measured without significant losses in strength and hardness.

Table 1. Composition, processing parameters and mechanical properties of new nanocrystalline ds-titanium materials (* 3-point bending test)

	Ti/1Si	Ti/1.5C	Ti/HS
alloying element composition			
Si [wt.-%]	1	-	0.5
C [wt.-%]	-	1.5	0.65
milling parameters			
rotation speed [rpm]	150	150	150
milling time [hrs]	64	20	64
thermal treatment in high	-	30 min	30 min
vacuum	-	400°C	400°C
SPS parameters			
pressure [MPa]	80	80	80
heating rate [K/min]	100	100	100
sintering temperature [°C]	700	700	700
sintering time [min]	6	6	6
bulk composition			
Ti_5Si_3 dispersoids [Vol.-%]	4.2	-	2.1
TiC_X dispersoids [Vol.-%]	-	9.2	4.0
Ti matrix [Vol.-%]	rest	rest	rest
mechanical properties			
vickers hardness [HV0.5]	392	348	320
*yield strength [MPa]	1425	1135	1310
*bending strength [MPa]	1650	1615	1930
*plast. elong. at break [%]	0.5	2	9.6

A pin-on-disc experimental set-up was used to investigate the wear behaviour of the new developed materials in comparison to c.p.Ti and TiAl6V4 as pin materials against TiAl6V4 as the material for the rotating disc.

$$k = \frac{A \cdot \Delta l}{F \cdot s} \qquad (1)$$

The wear coefficient k for each pin was calculated using equation (1) where A is the cross section area of the pin, l is the decrease of the pin length, F is the contact force between the pin and the disc and s is the total sliding distance. The wear curves in Fig. 2 show clearly an improvement in wear restistance by the new nanocrystalline and dispersion strengthened titanium materials Ti/1Si, Ti/1.5C and Ti/HS in comparison to c.p.Ti and also to the implant alloy TiAl6V4.

Consistant with the wear curves the wear coefficients of the new materials show no significant differences amongst each other (Table 2). Their wear coefficient is about five times better then c.p.Ti and twice as good as the implant alloy TiAl6V4.

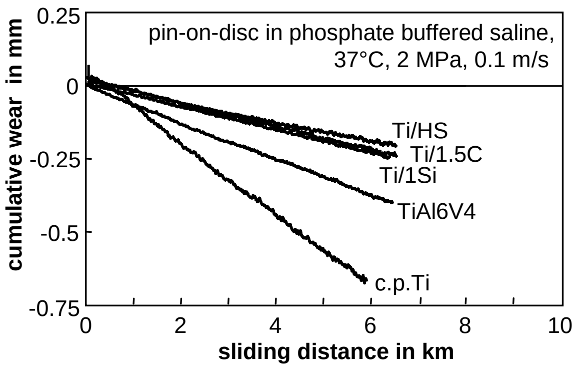

Fig. 2. Wear curves of the Ti/1Si, Ti/1,5C, Ti/HS, TiAl6V4 and c.p.Ti tested against a TiAl6V4.

Table 2. Wear coefficients of the testet materials in mm^3/Nm

c.p.Ti	TiAl6V4	Ti/1Si	Ti/1.5C	Ti/HS
$5.5 \cdot 10^{-5}$	$2.3 \cdot 10^{-5}$	$1.1 \cdot 10^{-5}$	$1.2 \cdot 10^{-5}$	$1.0 \cdot 10^{-5}$

3. Summary

Ultra-fine grained and dispersion-strengthened titanium materials manufactured by high energy ball milling followed by a compaction via SPS show a high strength, hardness and wear resistance. Use of the organic fluid as carrier of the dispersiod forming elements led to a significant ductility increase in the material which is supposedly caused by the finer and more homogeneously distributed dispersoids.

4. References

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