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Effect of Temperature and Pressure on the Densification of Titanium Silicide Compound

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Abstract

Titanium silicide compound was synthesized from the mixture of titanium and silicon powder with atomic ratios of 60:40 and 70:30. The powders were mixed by ball milling and pressed by different methods: by using uniaxial pressing at 64 MPa, cold isostatic pressing (CIP) at 200 MPa, hot forging (HF) at 648 MPa, and hot pressing (HP) at 24 MPa. The samples were then sintered at 1300°C or 1600°C for 2 hours soaking time in argon atmosphere. The sintered samples were subsequently characterized for phase constitution using X-ray diffraction (XRD). Ti_5Si_3 was observed as main compound of mixtures with both ratios - 60:40 and 70:30. Archimedes' method and scanning electron microscope (SEM) were used to measured density and investigate the microstructure of the sintered samples. It was found that the sample prepared from the mixture with ratio 70:30 has higher density than the sample of ratio 60:40 for all applied pressures. The density of samples prepared from the mixture of 70:30 and 60:40 sintered at 1300°C are in the range of 53-60% and 42-55%, respectively. It was found that densities of all samples sintered at 1300°C were not much different when higher forming pressure was applied by using CIP and HF. This means that pores are created during sintering. However, the microstructure of samples formed by CIP and HF showed some big pores inside the sample body while the sample formed at lower pressure by uniaxial press showed a more uniform pore size. By increasing the sintering temperature to 1600°C, the density of the uniaxial pressed sample was increased to 85%, and the pore size shrinks in comparison to the one sintered at 1300°C. Applying a lower pressure during sintering the sample at 1600°C by hot pressing, on the other hand, can produce a high density sample of 99% with a small amount of small closed pores.

Key words: Titanium silicide, Cold isostatic press, Hot forge**,** Hot press

Introduction

Titanium silicide compound such as $TiSi_3$, $TiSi_2$, TiSi, Ti_5Si_4 and Ti_5Si_3 can be prepared from various ratios of titanium and silicon metal. Among these silicide compounds, Ti_5Si_3 is known as an intermetallic compound which is suitable for high temperature applications due to the properties of a high melting point (2130°C), moderate density (4.32 g/cm^3), high temperature oxidation resistance, high hardness (11.3 GPa) and high Young's modulus (225 GPa).[1-2] Titanium silicide can be prepared by a variety of powder techniques such as hot pressing, hot isostatic pressing, reactive sintering, mechanical alloying, and thermal or plasma spraying.[3-6] Due to the limited fracture toughness of Ti_5Si_3 at room temperature, most researchers have paid attention to produce multiphase in Ti_5Si_3 compound by addition of Al, C, Ni, or Nb.[4, 6-7] However, research works on densification of Ti_5Si_3 dependence on pressure and temperature are limited. In this paper, varieties of pressure and temperature were applied to prepare Ti_5Si_3, and then their densities and microstructures were observed.

Materials and Experimental Procedures

Ti powder (99.7% purity, average particle size < 45μm) and Si powder (99.7% purity, average particle size < 45μm) were mixed in atomic ratio of 60:40 and 70:30 for 20 hours in Ar gas atmosphere. The mixed powder was compacted into specimens with 2 cm diameter and 0.5 cm

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thickness using varied pressures of 64 MPa (uniaxial pressing), 200 MPa (cold isostatic pressing, CIP), 648 MPa (hot forging, HF) and 24 MPa (hot pressing). The uniaxial pressed specimens were sintered at temperatures varied from 1100 to 1600°C while other specimens were sintered at 1300 and 1600°C in argon atmosphere. The heating rate and soaking time were 15°C/min and 2 hours respectively. The sintered specimens were measured for density by Archemidis method. X-ray diffractometer (XRD) and Scanning electron microscope (SEM) were used to determine the phase constitution and microstructure of the sintered specimens, respectively.

Results and Discussion

The sintered Ti: Si specimens of 60:40 and 70:30 were characterized for phase constitution by XRD as patterns show in Figures 1 and 2, respectively. The results reveal that single phase of Ti_5Si_3 was formed in the 60:40 specimens sintered at temperatures between 1100 and 1500°C; and at a high temperature of 1600°C, TiC was observed together with Ti_5Si_3 phase. On the other hand, TiC can be formed together with Ti_5Si_3 in 70:30 specimens sintered at a temperature range of 1100-1500°C. At 1600°C, this TiC phase will transform to Ti_3SiC_2. The TiC and Ti_3SiC_2 phases in specimens could originate from the diffusion of carbon inside the furnace into specimens during sintering process. Higher content of Ti in the specimen causes easier forming of TiC at low temperature, and TiC will react with some Ti_5Si_3 to form Ti_3SiC_2 at high temperature.

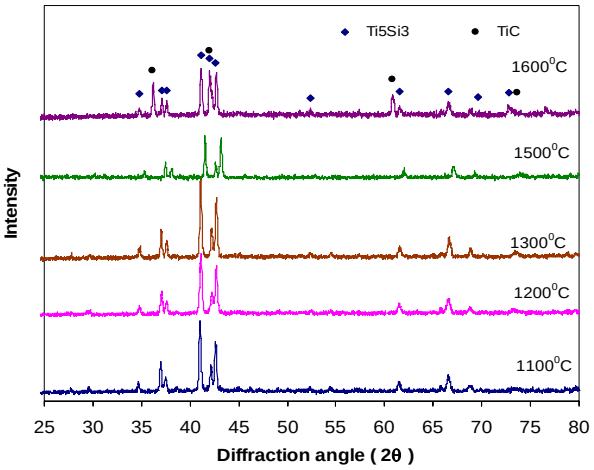

Figure 1. XRD patterns of the Ti:Si mixture of 60:40 sintering at different temperatures.

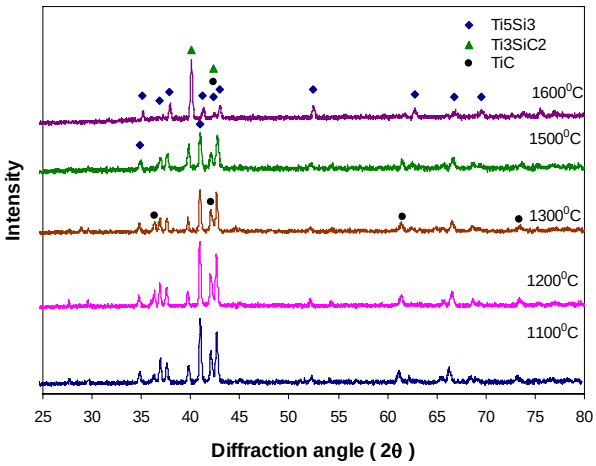

Figure 2. XRD patterns of the Ti:Si mixture of 70:30 sintering at different temperatures.

Table 1 shows density of specimens formed at different pressures after sintering at 1300°C. It was found that increasing the forming pressure could slightly increase the density of specimens after sintering. The specimen with high Ti content yields a higher density than that of lower Ti content, which agrees with the SEM micrographs in Figure 3. Moreover, Figure 3 shows that the specimens formed at higher pressure have bigger pore sizes than those formed at lower pressure. These phenomena can be explained by the fact that the Si vapor is trapped inside specimens that have been compacted at high pressure prior to sintering. On the other hand, Si vapor generated from specimens with lower forming pressure can easily move out during sintering before densification, and that results in smaller pores.

According to Tables 1 and 2, the density of the specimen formed at 64 MPa increases significantly from 57% to 86% when the sintering temperature is raised from 1300°C to 1600°C. Furthermore, by simultaneously applying low pressure (24 MPa) and heating (1600°C) the density of specimens can be raised to 99%, which is shown in Figure 4. This means that temperature is more effective to generate increase in density than pressure; however, applying pressure during sintering is most effective to obtain high densification.

Table 1. Density of Ti:Si mixtures of 70:30 and 60:40 ratios formed at various pressures and sintered at 1300°C.

Ti:Si	Bulk density		Apparent density		(Bulk density/ Apparent density) x100	
	60:40	70:30	60:40	70:30	60:40	70:30
Uniaxial press (63.69 MPa)	1.89	2.49	4.37	4.35	43.24	57.24
CIP (200 MPa)	1.79	2.33	4.27	4.36	41.92	53.44
HF (648MPa)	2.38	2.63	4.31	4.40	55.22	59.77

Table 2. Density of TiSi mixture of 70:30 ratio sintered at 1600°C.

Ti:Si	Bulk density	Apparent density	(Bulk density/ Apparent density) x100	%Porosity by Archimedes method
Uniaxial press (63.69 MPa)	3.70	4.31	85.84	14.16
Hot press (24.24 MPa)	4.34	4.40	98.63	1. 37

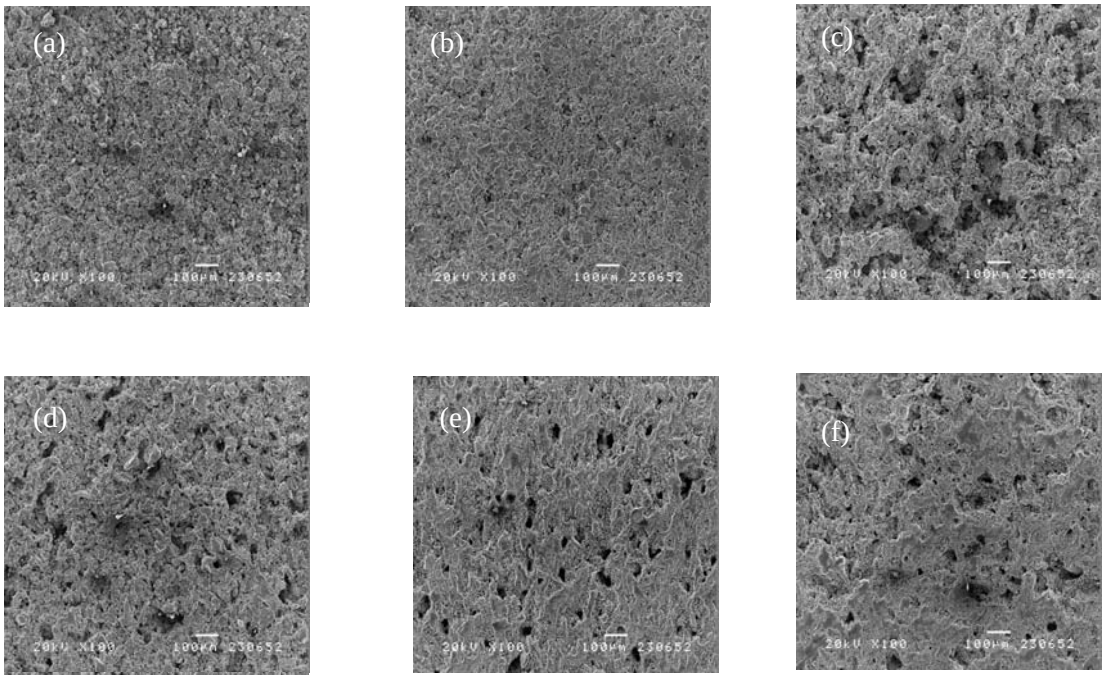

Figure 3. SEM micrographs of samples with Ti:S mixture of (a) 60:40 (uniaxial Press 64 MPa), (b) 70:30 (uniaxial Press 64 MPa), (c) 60:40 (CIP 200 MPa), (d) 70:30 (CIP 200 MPa), (e) 60:40 (HF 648 MPa), (f) 70:30 (HF 648 MPa).

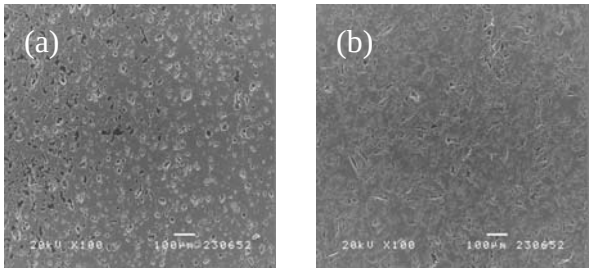

Figure 4. SEM micrographs of TiSi mixture of 70:30 sintered at 1600°C, formed by (a) uniaxial press at 64 MPa and (b) hot press at 24 MPa

Conclusions

Ti_5Si_3 can be synthesized from Ti and Si powders (70:30 and 60:40) sintered at 1100-1600°C. During sintering Si vapor can create, and form, the pores inside the specimen; it also retards the densification. Increasing forming pressure is insignificant in densification during sintering. On the other hand, applying lower pressure during sintering can remarkably enhance the densification of Ti_5Si_3 specimens.

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