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SELF-CONSOLIDATION MECHANISM OF Ti_5Si_3 COMPACT OBTAINED BY ELECTRO-DISCHARGE-SINTERING DIRECTLY FROM PHYSICALLY BLENDED Ti-37.5 AT.% Si POWDER MIXTURE

Characteristics of electro-discharge-sintering of the Ti-37.5at.% Si powder mixture was investigated as a function of the input energy, capacitance, and discharge time without applying any external pressure. A solid bulk of Ti_5Si_3 was obtained only after in less than 129 μsec by the EDS process. During a discharge, the heat is generated to liquefy and alloy the particles, and which enhances the pinch pressure can condensate them without allowing a formation of pores. Three step processes for the self-consolidation mechanism during EDS are proposed; (a) a physical breakdown of oxide film on elemental as-received powder particles, (b) alloying and densifying the consolidation of powder particles by the pinch pressure, and (c) diffusion of impurities into the consolidated surface.

Keywords: Sintering, Mechanical alloying, Powder consolidation, Intermetallic compounds, Titanium silicide

1. Introduction

Syntheses of intermetallic compounds with high melting points *via* mechanical alloying (MA) have been attempted in numerous studies [1,2]. In general, combustion reactions have been initiated by ball-milling in a variety of highly exothermic reaction mixtures. The formation of intermetallics from their elemental components were obtained during ball-milling due to its a self-sustaining high temperature reactions [3,4]. Among intermetallic compounds, Ti_5Si_3 has received more attention in high temperature structural materials applications owing to its low temperature toughness, high temperature strength, creep resistance, oxidation resistance, and relatively low density [5,6].

It has reported that a solid bulk typed Ti_5Si_3 was made by reacting with the stoichiometric mixture of Ti and Si powders at higher temperature or arc melting of Ti and Si pieces [7,8]. In spite of their research significance, relatively few studies on the sintering of Ti and Si powders have been investigated in recent years. The normal sequences in powder metallurgy operations were to compact a metal powder in a die at room temperature and subsequently sintered it at elevated temperatures. Not only were high pressure, high temperature, and long processing times required but, in the case of reactive materials, such as Ti and its alloys, an inert atmosphere were also inevitably required. The high temperatures involved in these processes, however, resulted in detrimental changes in both the microstructure and mechanical properties.

Lee and co-workers have reported that Ti and Ti-6Al-4V powders can be successfully consolidated into fully porous or solid bulk types without detrimental changes in both the microstructure and mechanical properties by employing an electro-discharge-sintering (EDS) technique [9-11]. However, the formation of Ti_5Si_3 solid compact by EDS has not yet been reported.

The EDS characteristics were reported as a function of the input energy, capacitance, and discharge time. The mechanisms of the EDS consolidation of the Ti and Si powder particles were given to produce a solid Ti_5Si_3 compact.

2. Experimental

The mean powder particle sizes of as-received Ti and Si were 45.0 μm and 8.0 μm, respectively. The purity of the powders was better than 99.95%. Ti and Si powders at stoichiometric ratio of 5:3 (Ti-37.5 at.% Si) were physically mixed for 10 hours in a ball mill for the preparation of reactant mixture.

Elemental powder mixture of 0.34 gram was vibrated into a quartz tube with an inner diameter of 4.0 mm that had a tungsten electrode at the bottom and top. A capacitor bank of 300 μF was charged with three different electrical input energies (2.5, 3.0 and 5.0 kJ). The charged capacitor bank instantaneously discharged through the powder column by on/off high vacuum switch which closes the discharge circuit. The voltage and current

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that the powder column experiences when the circuit is closed were simultaneously picked up by a high voltage probe and a high current probe, respectively. Outputs from these probes are fed into a high speed oscilloscope that stores them as a function of discharge time. The overall consolidation process by the discharge is referred to as electro-discharge-sintering (EDS).

The phase composition analyses for the reactant powder mixture and electro-discharge-sintered (EDSed) compacts were investigated by X-ray diffraction (XRD) using Cu K_α radiation. Each sample was sliced every two millimeters and the resulting cross-sections were examined under scanning electron microscopy (SEM).

3. Results and discussion

The Ti-37.5 at.% Si powder mixture was consolidated by a conventional hot-pressing process at 1200°C in a vacuum pressure of 2×10^{-6} torr by applying a pressure of 10 tons for two hours. As shown in (Fig. 1a), as expected, the morphology of the SEM micrographs revealed that the consolidation process did not successfully produce the compact in a bulk type, resulting in the formation of a porous structure with high surface areas. The surface morphology of the SEM micrograph of the cross section view of EDSed compacts at the input energy of 5.0 kJ is shown in (Fig. 1b). The compact was composed of powder particles that

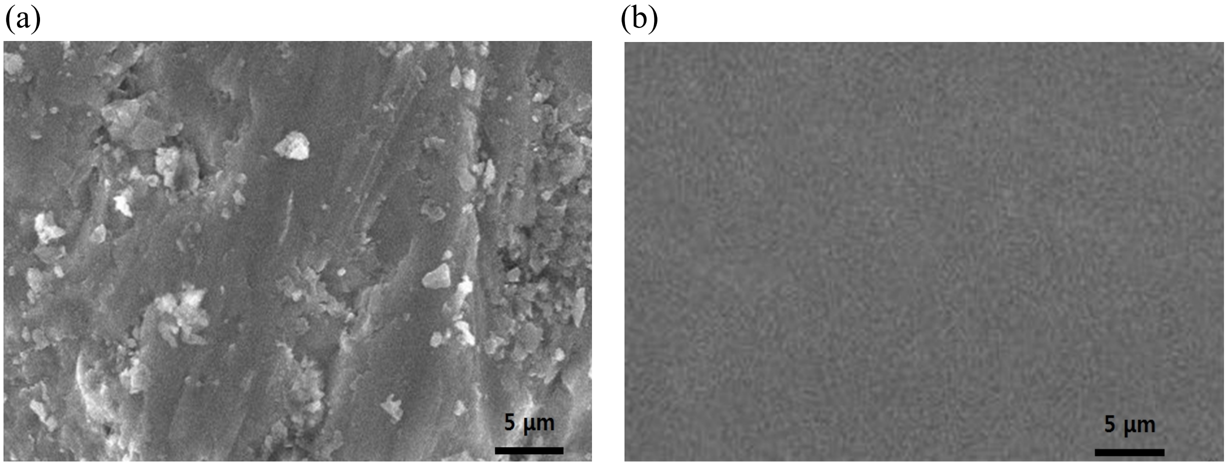

Fig. 1. SEM micrographs of the cross-sections of consolidated Ti_5Si_3 compacts obtained by (a) hot-pressing at 1200°C in a vacuum pressure of 2×10^{-6} torr by applying a pressure of 10 tons for two hours and (b) electro-discharge-sintering at the input energy of 5.0 kJ

were completely casted by the EDS. The density of the compact is approximately ~99% of theoretical value.

XRD patterns for the EDSed compacts as a function of input energy are shown in Fig. 2. The results indicate that peaks, corresponding to the phase of Ti_5Si_3, have been found, which lead to the fact that the Ti and Si powder mixture were simultaneously

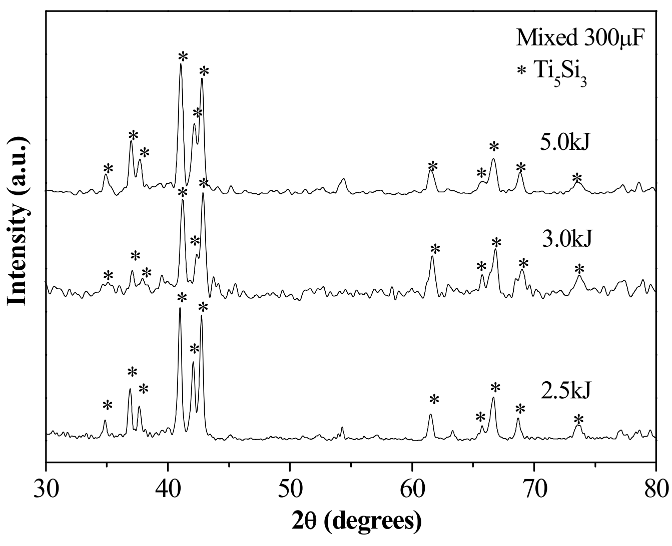

Fig. 2. XRD patterns for the EDSed compacts as a function of input energy

alloyed and casted into the unique phase of Ti_5Si_3 during EDS processing. The average crystallite size of EDSed Ti_5Si_3 compacts was determined as 120-151 nm by using Suryanarayana and Grant Norton's formula [12].

To investigate the consolidation mechanism of Ti_5Si_3 solid compact by EDS, electrical discharging characteristics were considered in terms of input energy and capacitance. A typical discharge curve (Fig. 3a) shows voltage and current in terms of discharge time. From the results shown in (Fig. 3a), the power (watt) curve is plotted in (Fig. 3b) against the discharge time. The discharge times for the duration of the first cycle at three different input energies are identical to be approximately 129 µsec. The amount of heat generated (DH) during a discharge can be obtained by using Eq. 1.

$$\Delta H = \sum[i^2(t)R(t)\Delta t] \tag{1}$$

Typical discharge characteristics are tabulated in Table 1 in terms of peak current, peak voltage, discharge time, and ΔH.

As a usual sintering process, the consolidation of metal powder requires a heat. To understand the effects of ΔH as one of discharge characteristics for the consolidation process, the temperature rise (ΔT), which is caused by the input energy, is now considered and estimated using Eq. 2:

$$W = mCp\Delta T \tag{2}$$

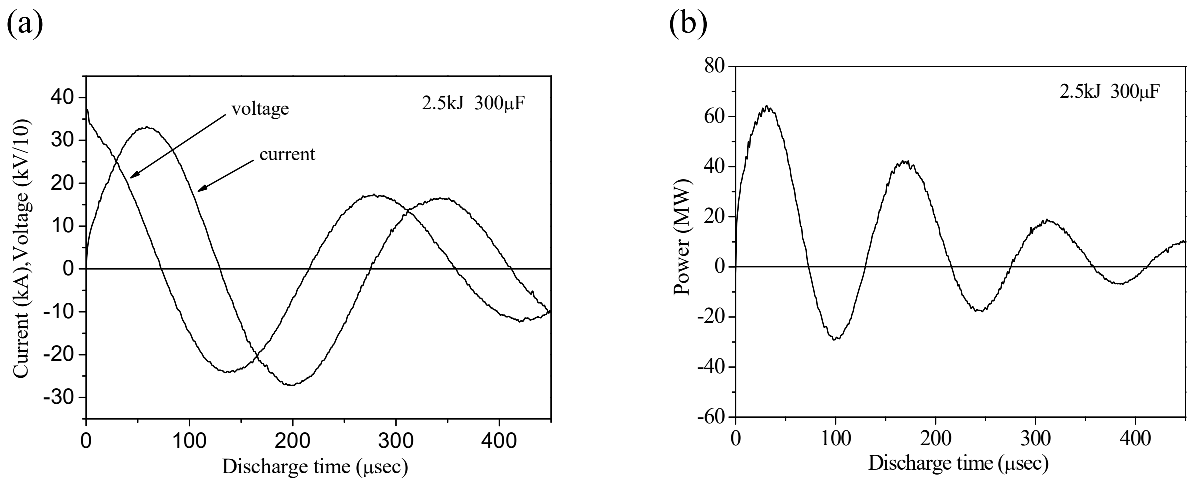

Fig. 3. (a) Typical discharge curve measured current and voltage on oscilloscope and (b) typical power curve versus discharge time (discharge condition: 300 μF, 2.5 kJ)

where m is the mass of the Ti-37.5 at.% Si powder and *Cp* is the specific heat of Ti_5Si_3. The electrical input power (*W*) was calculated by integrating current and voltage as a function of discharge time. The resulting data for the heat generated by EDS are listed in Table 2. It can be known that the EDS produce the heat significantly greater than the melting temperature of Ti_5Si_3. Such a heat generated is supposed to be high enough to vaporize the powder mixture. However, the duration of the heat rise as 129 μsec could be too short for the complete vaporization process, resulted in the phase transformation into a liquid. During EDS, the force which can pressurize the liquidus powder can be considered as a function of input energy.

TABLE 1

Peak voltage, peak current, discharge time, and heat generated (ΔH) during a discharge

Input energy (kJ)	Peak voltage (kV)	Peak current (kA)	ΔH (J)	Discharge time (μsec)
2.5	3.8	33.6	4181	129.4
3.0	4.1	36.0	4947	129.6
5.0	5.2	48.8	8572	129.8

TABLE 2

Temperature rise (ΔT), current density (j), and pinch pressure (P) produced by an electrical discharge

Input energy (kJ)	Current density (A/m^2)	Temperature rise (°C)	Pinch pressure (MPa)
2.5	4.34×10^{11}	2004	1371
3.0	4.66×10^{11}	3211	1582
5.0	6.31×10^{11}	5038	2900

When a capacitor bank is discharged through a powder column, a long cylindrical metal powder column conducting an axial current tends to contract radially inwards. At this moment the magnetic field generated by the current flow causes a diametric contraction, which is known as the pinch effect [13]. The magnitude of the magnetic field (B) can be obtained by using Eq. (3) :

$$B = {}^1\!/_2\,\mu r j \tag{3}$$

where μ is the permeability, j is the current density, and r is the distance from the center of the powder column. The resulting pinch pressure (P) is the mechanical force acting on the powder column that will produce a solid core. The pinch pressure is given by Eq. 4 :

$$P = \mu j^2 (a^2 - r^2)/4 \tag{4}$$

where a is the radius of the cylindrical powder column. The Ti-37.5 at.% Si powder particles are considered to be stacked in such a linear manner, resulting in the parallel straight current passage. The geometrical parameters needed for estimating the pinch pressure can be obtained and are tabulated in Table 3. Using these parameters, the maximum pinch pressure can be estimated in the center of the contact area (at $r = 0$). The resulting pinch pressures calculated under current experimental conditions are also listed in Table 3. It can be known that the pinch pressures between 1371 and 2900 MPa by the discharge were generated and could pressurize the liquidus powder particles, producing a bulk-typed Ti_5Si_3 compact without containing pores.

TABLE 3

Geometric parameters in the pinch pressure calculation

Number of particles on cross-section of powder column	6.67×10^4
Contact area of particle (m^2)	1.13×10^{-12}
Mean cross-sectional area of powder particle (m^2)	7.73×10^{-8}
Radius of the powder column (m)	1.2×10^{-4}

Since the pinch pressure is maximal at the center of powder column, its distribution across the cross-section of the compact should be different. The distribution of pinch pressures generated across the powder column is shown in (Fig. 4). It is very logical that the heat generated is the required parameter to melt the Ti-37.5 at.% Si powder particles and the pinch pressure can condense them without allowing the formation of pores across the compact.

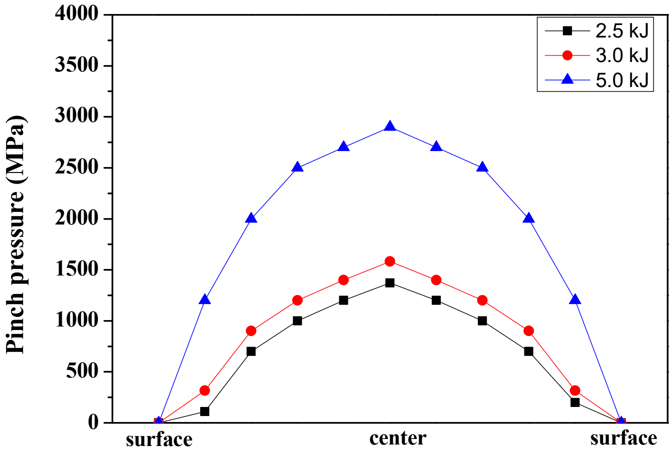

Fig. 4. Distribution of pinch pressure generated on the cross-section of electro-discharge-sintered Ti_5Si_3 compact measured at various input energies

Figure 5 shows the resistance change through the Ti-37.5 at.% Si powder column during EDS, which was determined from the recordings of voltage and current. It can be seen that there are four distinct regions. In stage I, electronic and physical breakdown of the oxide layer of Ti-37.5 at.% Si powder occurred, causing the rapid drop of resistance. In stage II, the resistance decreased very slowly. The heat generated during a discharge

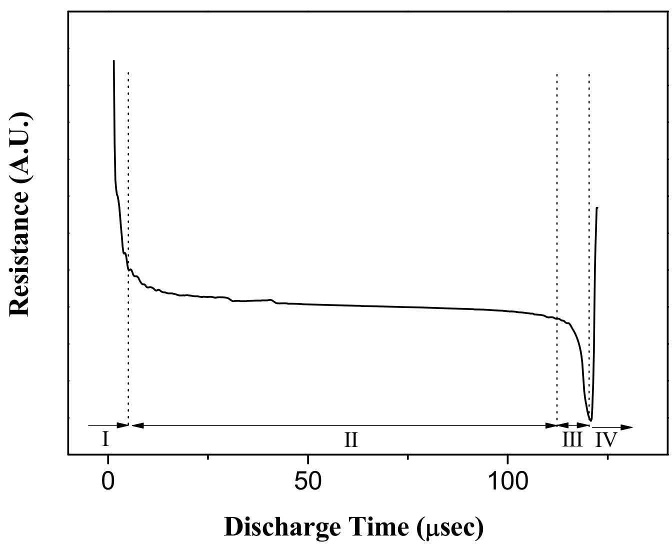

Fig. 5. Typical resistance variation of Ti and Si powder column calculated from the voltage and current recordings during an electro-discharge-sintering

would liquefy the Ti-37.5 at.% Si powder. Both condensation and densification of melted powder are promoted by the pinch force. In stage III, another rapid drop of resistance occurred due to the formation of solid. The rapid cooling occurs in this stage, resulting in the preservation of nano-sized crystallite of Ti_5Si_3 compact. In stage IV, the resistance increased very sharply which can be due to the surface modification by the diffusion process.

4. Conclusions

Electro-discharging-sintering using input energies of 2.0, 2.5 and 5.0 kJ through the Ti-37.5 at.% Si powder mixture were carried out without applying any external pressure. The reactant powders were successfully alloyed and consolidated into a solid bulk in the form of Ti_5Si_3 in less than 129 μsec. It is proposed that in the initial part of a discharge, physical breakdown of the oxide film of powder mixture occurs first and then alloying and consolidation processes follow, producing the bulked compact mainly composed of one phase of Ti_5Si_3 with the aids of heat generated and pinch pressure. Once the consolidation process is completed, the ambient impurities such as oxygen and carbon diffuse into the surface of the compact in the last stage of a discharge.

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