Article

Characteristics of the surface coating layer obtained by shock compaction and reaction synthesis through underwater shock compression

Kazuyuki Hokamoto*†, Sang-Hoon Lee**, Jung-Suk Lee*, Shigeru Tanaka**, Akihisa Mori**, Ryuichi Tomoshige***, and Masahiro Fujita***

*Shock Wave and Condensed Matter Research Center, Kumamoto University, 2-39-1 Kurokami, Kumamoto 860-8555, JAPAN

†corresponding author: hokamoto@mech.kumamoto-u.ac.jp

**Graduate Student, Graduate School of Science and Technology, Kumamoto University, JAPAN

***Faculty of Engineering, Sojo University, Kumamoto 860-0082, JAPAN

Received: May 27, 2005 Accepted: June 22, 2005

Abstract

The possibility for making thin coating layer through shock compaction and reaction synthesis from some mixed elemental powders is discussed, and the microstructure is characterized in the present investigation. Underwater shock wave in the order of 1-15 GPa derived from the detonation of an explosive was employed for the reaction. Mg plus Si to form Mg_2Si and Ti plus Si to form Ti_5Si_3 were selected as coating materials. After shock compression, the compacted layer was successfully recovered without voids and cracks. Partial or no reaction was observed in the coating layer after shock compaction, and the followed heat treatment made possible to form reacted intermetallic compound in the case of synthesizing Ti_5Si_3.

Keywords: Shock compaction, Shock synthesis, Coating, Underwater shock wave

1. Introduction

Shock compaction and shock synthesis have been investigated intensively for making various bulk materials[1]. So far, some attempts have been made by some of the researchers[2)~4)]. The making of crack-free samples is a key issue of the related research area and the use of underwater shock compression technique enables to decrease the number of cracks due to relatively long pressure duration[2)~4)].

The present investigation intends to suggest a new possibility of compressing thin powder layer on a substrate material[5]. The synthesis of intermetallic compounds is investigated in this study, and Mg plus Si to form Mg_2Si and Ti plus Si to form Ti_5Si_3 was selected as coating materials. Mg_2Si is known as wear and corrosion resistant material[6), 7)], and Ti_5Si_3 is expected for its use in high-temperature applications due to its high melting point and its excellent mechanical properties at high temperature[8), 9)]. The experimental results suggest that the powder compaction was successful but the shock-induced reaction was slightly difficult. The following heat treatment to induce reaction was also tried for some of the samples recovered.

2. Experimental assembly used for the experiments

For the experiments, two types of underwater shock compressing assembly were employed as shown in Fig. 1 and Fig. 2. The SEP explosive (produced by Asahi-Kasei Chemicals Corp., Japan) of density 1300 kg m^{-3} and detonation velocity 7 km s^{-1} was employed in all the experiments. The closed type assembly shown in Fig. 1 has been developed and used for shock compaction of powders, which is capable of generating relatively high underwater shock pressure in the order of 5-15 GPa[4]. The other open type one as shown in Fig. 2 was developed for underwater explosive welding[10), 11)], and the available pressure ranges from 1-3.5 GPa. In both the assemblies, the shock pressure

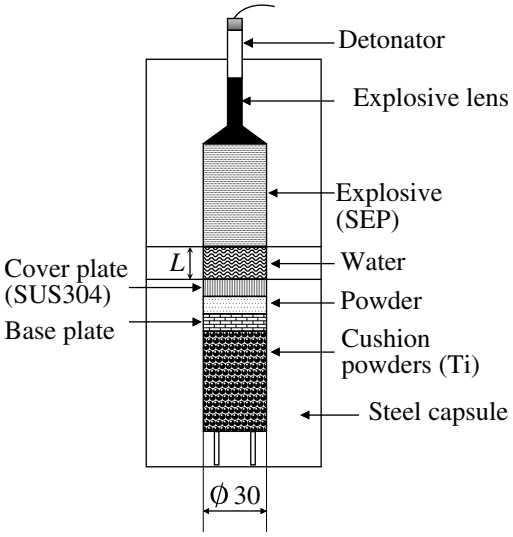

Fig. 1 Closed type assembly for shock compression experiments.

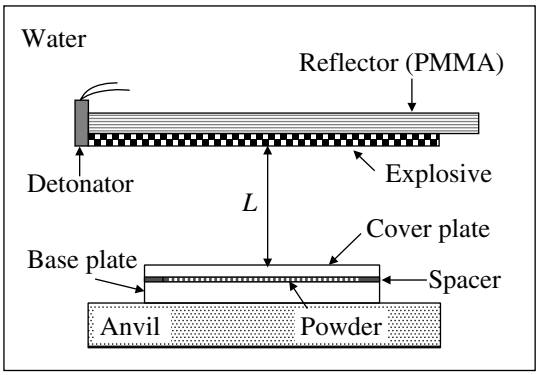

Fig. 2 Open type assembly for shock compression experiments.

can be regulated only by changing the height of water column *L*.

The solid line as shown in Fig. 3 shows the maximum underwater shock pressure Pm numerically calculated using AUTODYN-2D at a distance from explosive *L* for the open type assembly shown in Fig. 2. Parameters necessary for the calculation has been reported elsewhere[10]. Also, the pressure for the closed type assembly has been calculated[4] and is suggested as a dashed line in Fig. 3. The pressure profile numerically calculated at *L*=5 mm for open type assembly is shown in Fig. 4. After attaining a peak pressure, the pressure gradually decreased in 10 microseconds or more which is longer than the use of hypervelocity impact of a flyer plate as normally employed[1]. The same type of pressure profile can be obtained for closed type assembly though the order of maximum pressure is different[4]. Such pressures can give better results for recovering samples without defects[4].

The experiments conducted are listed in Table 1. Mg+Si powders were mixed for 5 h and Ti+Si powders were mixed for 3 h in Ar atmosphere using a high-energy ball mill P-7, made by Fritsch to mix these elemental powders mechanically. Some compaction experiments were also conducted for mechanically mixed Ti+Si powders further mixed with TiAl powders as to make a composite layer. The substrate was ground using abrasive powders (#400). Initial packing density of powders was fixed about 0.6 in all the experiments conducted and the powders were put uniformly on the substrate at a height of 300 μm in thickness for the experiments TS1 & 2 and TSTA1 & 2, and at 1 mm for MS1 & 2. Since we fixed *L*=5 mm for closed assembly and *L*=20 mm for open type assembly, the maximum underwater pressure Pm was estimated about 12 GPa and 1.7 GPa, respectively.

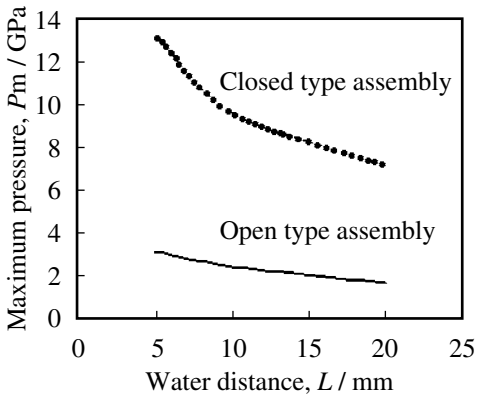

Fig. 3 Change in maximum underwater shock pressure with distance from explosive.

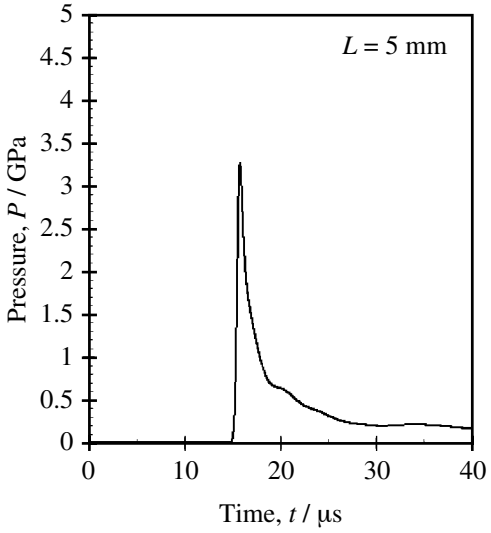

Fig. 4 Numerically simulated pressure profile where distance from explosive *L*=5 mm.

Table 1 List of experimental conditions.

No.	Assembly	Powders	Base plate	Cover plate	Main explosive	Results
TS1	Closed type (Fig. 1)	Ti:Si (5:3)*	Titanium 3 mm-thick	JIS SUS304 2 mm-thick	SEP 50 mm-thick	Fully recovered, no reaction
TS2	Open type (Fig. 2)			Aluminum 0.1 mm-thick	SEP 5 mm-thick	Fully recovered, no reaction
TSTA1	Closed type (Fig. 1)	Ti:Si(5:3)* (40 mass%) TiAl (60 mass%)		JIS SUS304 2 mm-thick	SEP 50 mm-thick	Fully recovered, no reaction
TSTA2	Open type (Fig. 2)			Aluminum 0.1 mm-thick	SEP 5 mm-thick	Fully recovered, no reaction
MS1	Closed type (Fig. 1)	Mg:Si (1:2)*	JIS AS31 (Mg alloy) 1mm-thick	JIS SUS304 2 mm-thick	SEP 50 mm-thick	Fully recovered, partial reaction
MS2	Open type (Fig. 2)	*atomic ratio		Aluminum 0.3 mm-thick	SEP 5 mm-thick	Partial recovery, partial reaction

▸ Titanium powder : -45 μm, Sumitomo Sitix Corp.
▸ TiAl powder : -150 μm, Sumitomo Sitix Corp.
▸ Silicon powder : -325 mesh, CERAC
▸ Magnesium powder : -20 mesh, Furuchi Chemical

3. Results and discussion

Most of the samples were recovered successfully as listed in Table 1. Under a moderate experimental condition as shown in the upper view (Fig. 5), no macroscopic cracks were observed and the powders were uniformly compressed to form a coating layer.

The cross-sectional view of the samples is shown in Fig. 6. As shown in the figure, the coating layer was fully densified and no micro-cracks were observed.

The enlarged microstructure of Mg+Si powders (sample number MS1) is shown in Fig. 7. The XRD patterns of the sample showed partial reaction to form Mg_2Si as confirmed in the microstructure. Due to the relatively large sized Mg powders commercially provided, the reaction was not induced uniformly due to the non-uniform mixing condition of the powders. Therefore, the original mixing condition should further be considered carefully as it is difficult due to the reactivity and the larger sized Mg powders. The Vickers hardness of the Mg powder part was 69.6 Hv and the hardness of the reacted part was 250 Hv as pointed by arrows.

Figure 8 shows the enlarged microstructure of Ti+Si powers (sample number TS1). The powders were fully compressed but the Ti and Si powders were not reacted as

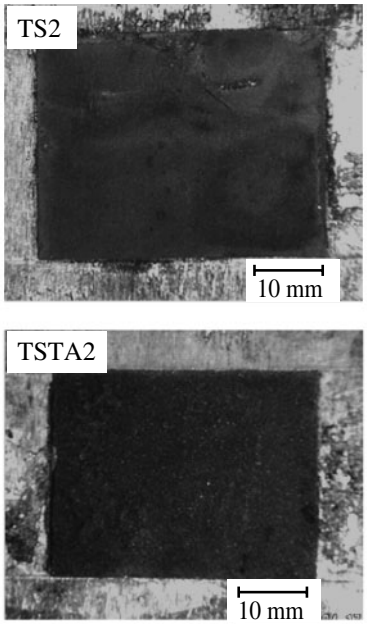

Fig. 5 Upper view of samples recovered (sample number TS2 and TSTA2).

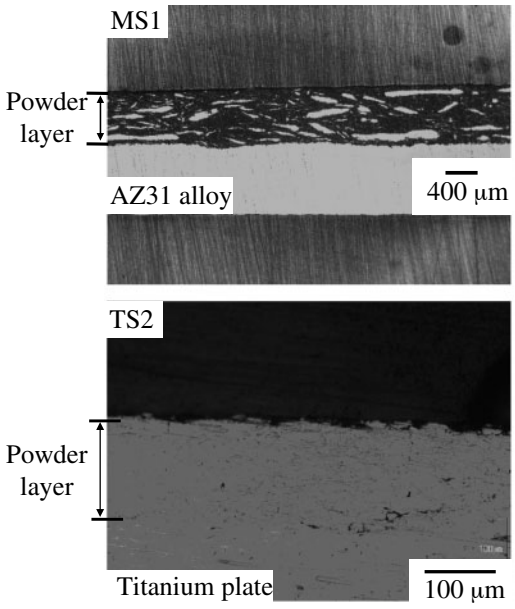

Fig. 6 Cross-sectional view of samples recovered (sample number MS1 and TS2).

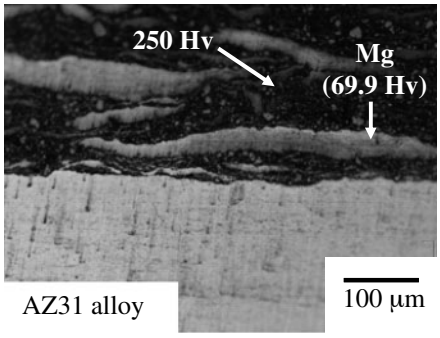

Fig. 7 Enlarged microstructure of Mg+Si powder layer (sample number MS1).

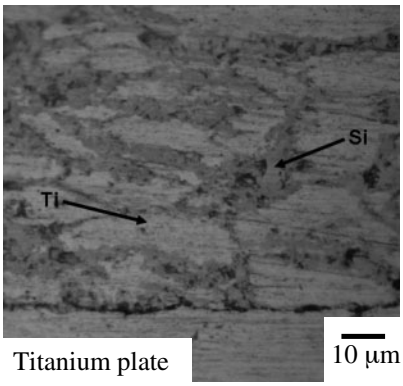

Fig. 8 Enlarged microstructure of Ti+Si powder layer (sample number TS1).

found in Fig. 8 and confirmed through XRD analysis.

As listed in Table 1, the results for closed and open type assemblies were almost similar though the pressurizing condition was quite different. Thinking about the pressure applied to the Ti+Si powders, it was confirmed that the pressure applied for closed type assembly is enough to induce reaction for making large-sized bulk materials[4], but the reaction was not induced in the present experiments. The reason should further be discussed in future but there is a possibility that the use of small amount of reactive powders may not ignite to induce reaction.

Since our final goal is to synthesize intermetallic compound, the following heat treatment at 1000 ˚C for 1 h in Ar atmosphere was conducted for Ti+Si samples (TS1, TSTA1). The synthesizing temperature was expected to decrease due to the activation through shock processing as reported earlier[4]. The Ti+Si sample (TS1) was not fully reacted still showing Ti peaks in XRD analysis. The Ti+Si and TiAl composite (TSTA1) was also heat treated under the same condition. The microstructure of the heat treated sample is shown in Fig. 9. The Ti_5Si_3 area suggested by an arrow was reacted showing high Vickers hardness 850Hv. It is slightly lower than the value measured in the former investigation[4], but still higher than the Ti_5Si_3 commercially available (300~400 Hv). Such a high hardness is due to a very fine substructure induced by shock processing and the following heat treatment as reported earlier[4]. Figure 10 shows the XRD pattern for the reacted part measured using micro-focus X-ray diffraction machine. It is confirmed that Ti_5Si_3 is synthesized through the following heat treatment. No separation of the coating layer through heating was generated but the formation of other intermetallic compound layer less than 10 μm in thickness was confirmed between the coated layer and a mild steel substrate. As far as the brittle intermetallic zone is thin, it does not harm the bonding strength much[12] but it should be considered carefully when the temperature or the time of heat treatment is increased. A thin reaction zone was also observed between the TiAl and Ti_5Si_3 parts, which is considered Ti_2Al (α' phase) as found in Fig. 10. Some areas of the mixed Ti+Si part of this sample were not reacted. It is considered that the initial mixing condition was still not enough and should further be mixed in the order of μm or less to make possible to induce reaction easily.

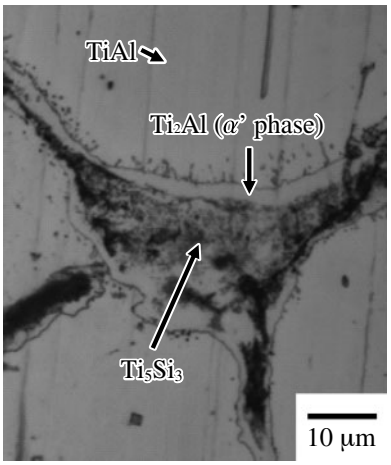

Fig. 9 Microstructure after heat treatment at 1000˚C for 1 h (sample number TSTA1).

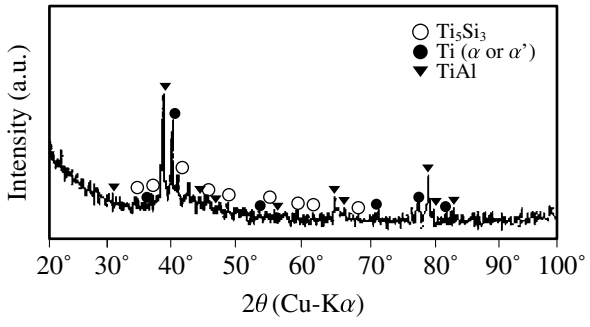

Fig. 10 XRD pattern for heat treated sample TSTA1 using micro-focus X-ray diffraction machine.

4. Conclusions

A new method of surface coating through shock compression and synthesis was proposed and the possibility for compacting a thin layer was suggested. Since the synthesis reaction through direct shock pressurization was not easy due to the limited amount of the powders used, the following heat treatment was performed for Ti and Si powder compacts, and the formation of intermetallic compound Ti_5Si_3 was confirmed as expected. High hardness in the synthesized Ti_5Si_3 intermetallic compound part was obtained due to the fine substructure through shock processing in a composite mixed with TiAl powders.

Acknowledgements

The supports by the 21st Century COE program "Pulsed Power Science" of Kumamoto University and the Grant-in-Aid for Scientific research from JSPS are gratefully acknowledged.

References

1) M. A. Meyers, "Dynamic Behavior of Materials", (1994), Wiley.
2) K. Hokamoto, S. Tanaka, M. Fujita, T. Kodama and Y. Ujimoto, Scr. Mater., 39, 1383 (1998).
3) K. Hokamoto, S. Tanaka and M. Fujita, Int. J. Impact Eng., 24, 631 (2000).
4) K. Hokamoto, J. S. Lee, M. Fujita, S. Itoh and K. Raghukandan, J. Mater.Sci., 37, 4073 (2001).
5) S. H. Lee, K. Hokamoto, J. S. Lee and Sh Khameneh Asl, Proc. ACHPR-2, Nara, Japan Nov.1-5, 2004, (2005) (Total 6 pages as CD-ROM).
6) K. Kondoh, H. Oginuma, T. Tsuzuki and T. Aizawa, Mater. Trans., 44, 611 (2003).
7) K. Kondoh, H. Oginuma, A. Kimura, S. Matsukawa and T. Aizawa, Mater. Trans., 44, 981 (2003).
8) S. A. Namjoshi and N. N. Thadhani, Scr. Mater., 40, 1347 (1999).
9) P. J. Counihan, A. Crawford and N. N. Thadhani, Mater. Sci. & Eng. A, 267, 26 (1999).
10) A. Mori, K. Hokamoto and M. Fujita, Mater. Sci. Forum, 465-465, 307 (2004).
11) K. Hokamoto, Y. Ujimoto and M. Fujita, Mater. Trans., 45, 2897 (2004).
12) K. Hokamoto, T. Izuma and M. Fujita, Metall. Trans. A, 24A, 2289 (1993).

水中衝撃圧縮による衝撃成形および衝撃反応合成によって得られる表面被覆層の諸特性

外本和幸*†，李　尚勲**，李　政錫*，田中　茂**，森　昭寿**，
友重竜一***，藤田昌大***

本研究では，いくつかの元素混合粉末に対する衝撃成形および衝撃反応合成の可能性について議論し，得られた材料の組織について検討が行われた。爆薬の爆轟によって得られる1-15 GPa程度の水中衝撃波が，粉末の成形・反応に用いられた。MgとSiからのMg_2Siの合成，TiとSiからのTi_5Si_3の合成が被覆のための対象材料として検討された。衝撃波負荷後，成形層はボイドやクラックを形成することなく良好に回収された。衝撃波負荷後の試料では，部分的反応もしくは未反応の成形体が被覆層として得られることがわかり，引き続いて行われる熱処理によってはじめて，反応生成物として金属間化合物であるTi_5Si_3を得ることが可能であることが判明した。

*熊本大学衝撃・極限環境研究センター　〒860-8555　熊本市黒髪2-39-1
†corresponding author: hokamoto@mech.kumamoto-u.ac.jp

**熊本大学大学院生　〒860-8555　熊本市黒髪2-39-1

***崇城大学工学部　〒860-0082　熊本市池田4-22-1