Article

Synthesis of various composite materials through underwater shock wave compression

Jung-Suk Lee*, Kazuyuki Hokamoto**, K.Raghukandan***, Shigeru Itoh**, Akira Chiba***, and Masahiro Fujita****

In the present investigation, various powders are shock compressed by underwater shock waves generated by the detonation of an explosive. Underwater shock synthesis using explosives offers unlimited potential in the otherwise difficult-to-synthesis metal powders. Metal matrix composites and composites of intermetallics, offering extra strength, stiffness and higher temperature capabilities in comparison to the conventional materials, are fabricated. The shock synthesis of composites of aluminum, titanium and titanium based intermetallics is addressed to in this paper. An experimental set-up, consisting of explosives, water column and powder parts, is employed for the present investigation. The explosive is detonated through an explosive lens to obtain a planar shock wave to ensure uniform consolidation. The shock waves propagating through the water medium can be controlled by the manipulation of the height of water column. This method is a one dimensional shock transmission technique and can be used for obtaining relatively low underwater shock pressure at 8 – 13 GPa. The synthesized compacts are subjected to microanalysis and the effects on intra-particle deformation, inter-particle interaction, fracture, and material configurational changes are discussed.

1. Introduction

During the past few decades the need to create advanced materials has increased manifold. Particularly so, in the areas of aerospace, electronics, medical and other related industries wherein improved and unique properties are mandatory. Shock loading offers a valuable alternative to the conventional processes. The application of shock loading on porous powder mixtures can result in the modification of the microstructure and create favorable conditions based on the drastic change of the state within a short period. Shock synthesis can be effectively used to create new materials, which can not be produced by conventional methods.

The present investigation explores a method for shock compressing powders using underwater shock wave. Using the assembly, it is easy to control the shock pressure by changing the dimensions of the assembly [1,2]. Controlled pressure using such technique is applied to some powder mixtures, and the possibility for making composites through deformation of softer component and through reaction of mixed element powders are demonstrated. The samples recovered are characterized using optical microscope, SEM and X-ray diffraction analysis.

2. The assembly and the experimental procedure

The assembly used for the experiments is illustrated in Fig.1. The assembly is composed of three parts, (1) explosive layer, (2) water layer and (3) powder layer, and the layers are contained in

Received : May 17, 2002
Accepted : November 21, 2002
*Graduate School of Science and Technology, Kumamoto University, Kumamoto 860-8555, JAPAN
TEL+81-96-342-3740 FAX+81-96-342-3729
e-mail jslee@mech.kumamoto-u.ac.jp
**Shock Wave and Condensed Matter Research Center, Kumamoto Univ., Kumamoto, JAPAN
***Dept.of Mech.Eng.& Mater.Sci., Kumamoto University, Kumamoto, JAPAN
****Dept.of Mech.Eng., Sojo University, Kumamoto 860-0082, JAPAN

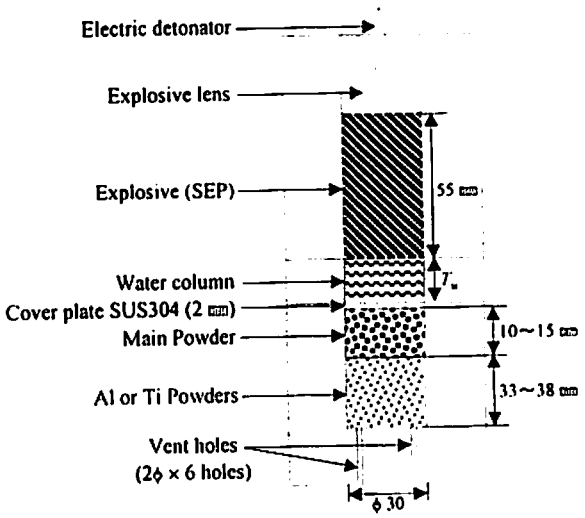

Fig. 1 Assembly used

special capsules made of mild steel. The main explosive used was SEP, produced by Asahi-Kasei Corp. (detonation velocity and the density are 7.0 $km \cdot s^{-1}$ and 1310 $kg \cdot m^{-3}$ respectively). The main explosive was detonated using explosive lens for obtaining planar waves to compress the powders uniformly. In comparison with the conventionally employed cylindrical method [3], the sample shows quite uniform condition of recovery [1,2]. The cylindrical method sometimes induces central hole due to a formation of mach stem [1] caused by converging high-pressure towards the center.

Figure 2 shows the simulated pressure distribution [5] in the water containing capsule, and the pressure is observed to reach the powder layers quite uniformly. The applied pressure can be regulated by varying the thickness of the water layer (t_w). By changing the thickness of water, t_w [1,2], the critical condition for reaction has been established for Ti and Si powder mixture to synthesize the intermetallic compound Ti_5Si_3. The variation of t_w from 5 mm to 15 mm reduced the peak water pressure from 12.7 GPa to 8.1 GPa [1,2]. The pressure range obtainable is not so high in comparison with the case for the compaction of diamond and related material powders [6], and this enables recovery of crack-free compacts.

Two kinds of experiments viz., (1) shock consolidation for making Al-base composites and (2) shock synthesis of intermetallic compound for making various composites, were conducted. Details of the experimental conditions are listed in Table 1. In case of the first kind of experiments industrial pure aluminum powders (>99.9 % and 100 – 200 mesh) and PAN based carbon short fibers (average 7 μm diameter and 200 μm long) were mixed using conventional ball-mill for 12 h. In case of the second set of experiments pure titanium (>99.4 % and 45 μm) and silicon (>99.5 % and – 325 mesh) powders were ball milled for 48 h, and then, mixed with other non-reactive powders. The

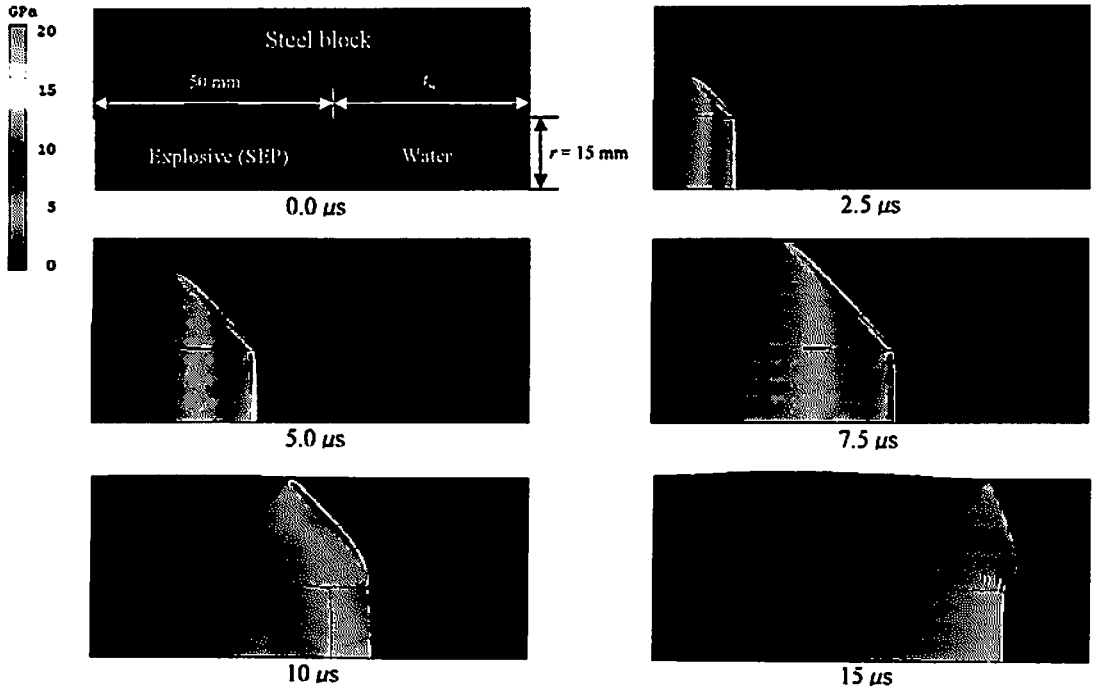

Fig. 2 Simulated pressure distribution in water container.

Table 1 Experimental conditions.

Experiment Number	Poeders(*atomic ratio)	Water thickness(mm)	Recovered condition
#AC1	Al (85vol%) + C_f (15vol%)	20	Densified, non-reacted
#TI1	Ti:Si (5:3*) (50mass%) + Ti (50mass%)	5	Fully densified, reacted
#TI2	Ti:Si (5:3*) (50mass%) + TiAl (50mass%)	5	Fully densified, reacted

※ TiAl powder (>99.8% and -150μm)

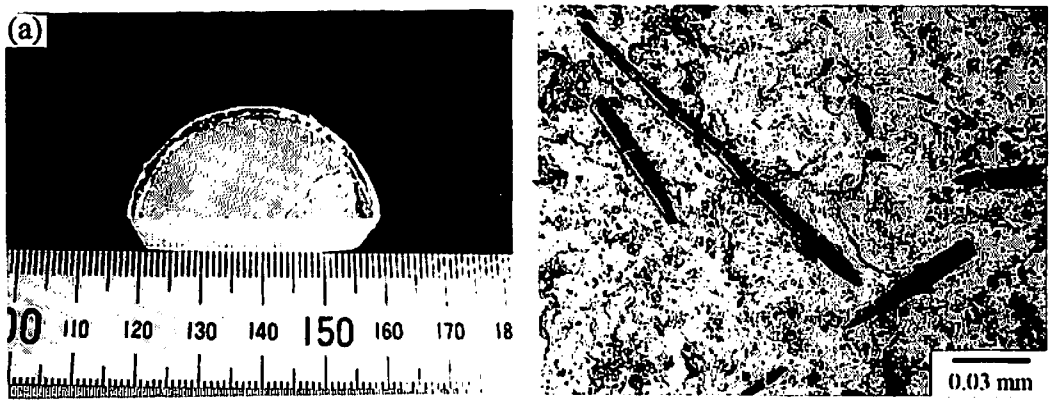

Fig. 3 Appearance (a) and polished surface (b) of aluminum-carbon short fiber composite obtained (#AC1).

main powders of the composite layer were stacked (10 – 15 mm) at approximately 60 % in the theoretical density, and a thick aluminum or titanium powder layer was provided beneath the main powder layer for alleviating the detrimental effect of reflected tensile waves and to facilitate easy recovery of the compacts.

3. Results and discussion

Figure 3 shows the appearance and the microstructure of polished surface of the recovered aluminum-carbon short fiber composite (#AC1). The whole sample of the compact is successfully recovered, and no macro- and microscopic cracks are observed in the cross-sectional area. Carbon fibers are well dispersed in aluminum matrix and the compact is fully densified by the plastic deformation of aluminum powders. The original surface of aluminum particle is unobserved and a strong interparticle bonding between the aluminum powders has been achieved under intensive deformation at an extremely high velocity. Though the carbon fibers are undeformed due to their poor ductility, the bonding between the aluminum and the fibers has been achieved through an intensive deformation of aluminum around the fiber surfaces. No trace of pulled-out fibers is found in the cross-sectional area, and the result suggests that the matrix-fiber bonding is strong enough to transfer the load applied and which is due to the strengthening of the composite by the fiber reinforcement. The measured average Vickers hardness under 19.6 N load is 808 MPa, which is quite higher than the pure aluminum (426 MPa).

The primary difficulty encountered is the fracture of the fibers during ball milling and during shock compression. The average aspect ratio l/d (l: length, d: diameter of the fiber) decreased after ball milling and after shock compression at 9.5 and 3.4 respectively. As to act as reinforcement, a higher aspect ratio is desirable and is expected to be made available by hand mixing. The experimental result without ball milling showed a compact of poor interparticle bonding. Agglomerated fibers harm the densification of the compact. For mixing the aluminum powders and the carbon short fibers, one possible way is to establish the optimum condition for mixing by changing the time or the method of mixing.

X-ray diffraction pattern of the recovered compact (#AC1) shows no evidence of the formation of aluminum carbide at the interface (Fig. 4). Figure 4 (b) shows weak unknown peaks close to

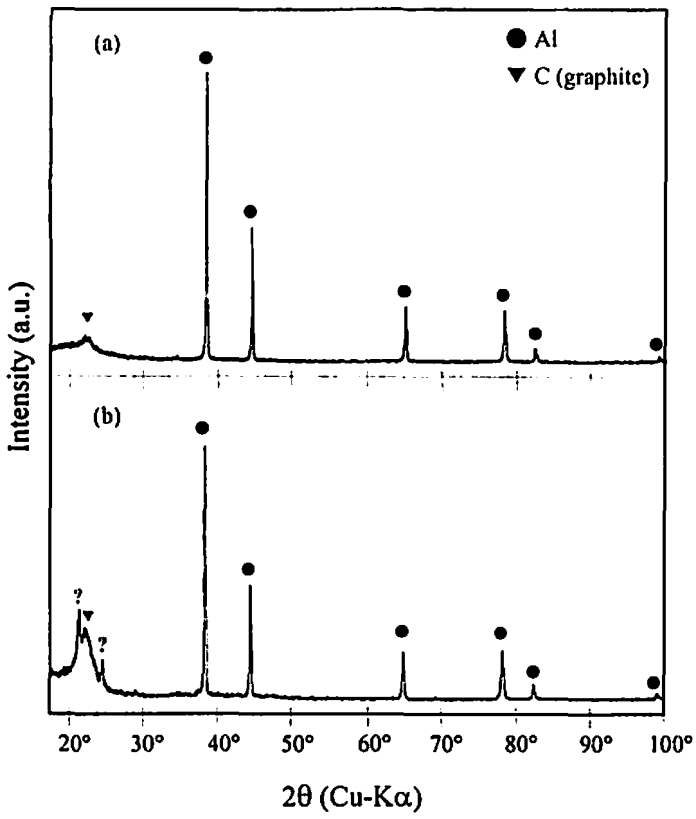

Fig. 4 X-ray diffraction patterns of as mixed (a) and after shock compressed (b) samples.

the peak of graphite, but these unknown peaks are sometimes measured in as ball mill mixed condition. The processing time, in the order of a few microsecond [1)2)], enables the recovery of the sample without reaction. The broadening of diffraction peaks of aluminum after shock compression shows refined crystalline size which is estimated based on the following equation [7],

$$t = \frac{0.9\lambda}{B\cos\theta_B} \qquad (1)$$

where t is crystallite size, λ is the wave length of the X-ray source, B is the full width of the diffraction peak at half maximum and θ_B is the diffraction angle. The crystalline size is 28 nm and the refined substructure in aluminum under an intensive deformation is evident.

For the mixture of titanium and silicon powders, some experiments to obtain their intermetallic compound are performed. The powers were mixed together first, and then, further mixed with the other non-reactive powders. Without using non-reactive powders, the exothermic reaction heat caused excessive melting which resulted in many defects like pores [1)2)]. The non-reactive powders act as heat sink to release the excessive heat from the reaction layer [8)9)]. Once the cooling time of the reacted layer is less than the time of pressurization, the whole sample is expected to be recovered successfully as reported by Kondo et al. [10] while discussing surface melting of powders during shock consolidation process. Figures 5 and 6 show the microstructure of the composites recovered, and the compacts are fully densified without defects in both cases. The mixed titanium and silicon powders are fully reacted, and the formation of Ti_5Si_3 is confirmed through X-ray diffraction analysis. The Vickers hardness of the reacted zone showed quite high value ranging from 9.2 – 11.5 GPa (under 0.245 N load) which is higher than the hardness of the commercially available Ti_5Si_3 [1)2)]. The crystalline size calculated using equation (1) was 36 nm, and the high hardness is attributed to the refined microstructure caused by the shock processing.

In the shock synthesized titanium-based

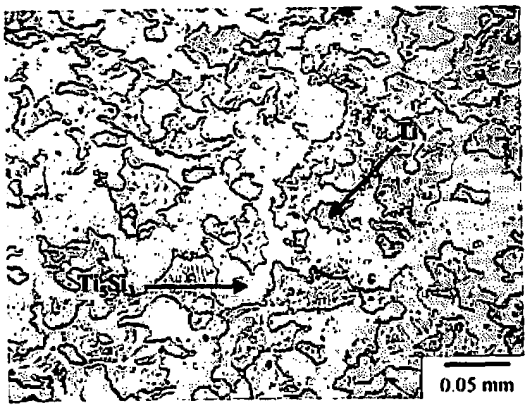

Fig. 5 Cross-section of Ti_5Si_3/Ti composite obtained (#TI1).

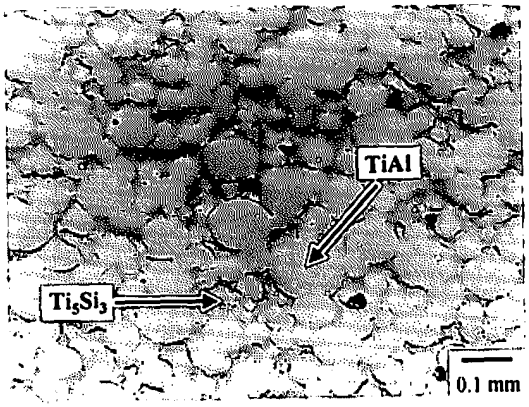

Fig. 6 Cross-section of Ti_5Si_3/TiAl composite obtained (#TI2).

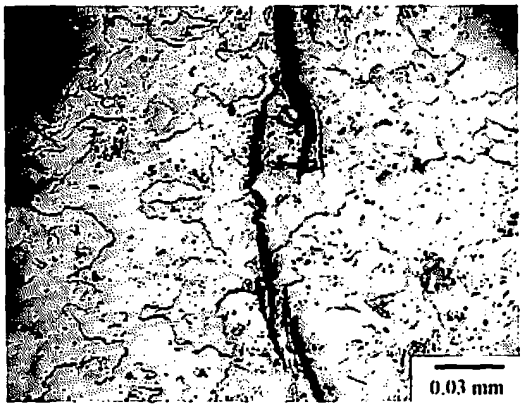

Fig. 7 Cracks appeared in Ti_5Si_3/Ti composite obtained (#TI1).

composites, few cracks were observed at periphery of the compacts and a propagated crack in the sample is shown in Fig. 7. Since the crack propagated in one direction and it is separated by intergranular fracture, it is concluded that the failure is a post synthesization phenomenon and is caused by the reflected tensile waves. The reduction of the formation of cracks can be affected by changing the dimension of the assembly so as to regulate the propagating shock waves and their reflection in the capsule.

Finally, high fracture toughness is expected both for aluminum-carbon fiber and titanium-based intermetallic composites not only by the existence of dissimilar phases but also the refined microstructure induced by the effect of a process at an extremely high-strain-rate. It is one of the important advantages of the process using shock compression. Further characterizations for the properties of the recovered composites are being performed.

4. Conclusions

Using an assembly of underwater shock compression, the possibility for making some composites is suggested both through shock consolidation of powders and through shock-induced reaction for generating intermetallic compound. The carbon short fiber reinforced aluminum composite shows increased hardness due to the strengthening by the carbon fibers, though the aspect ratio of the fibers observed to have decreased during processing. Composites of intermetallics are also fabricated through shock-induced reaction and the shock synthesized intermetallc Ti_5Si_3 shows quite high hardness in comparison with commercially available ones. Such high hardness is obtained due to the refined microstructure by shock processing. Superior mechanical properties are expected for such materials of having very refined microstructure. Shock compression may use as one of the techniques of refining the crystalline by an intensive deformation at an extremely high-strain-rate.

Acknowledgements

The authors acknowledge the help rendered by Mr. Susumu Akimaru, Shock Wave and Condensed Matter Research Center, Kumamoto University, for the conduct of experiments.

References

1) K. Hokamoto, J.S. Lee, Y. Suwa, S. Itoh, R. Tomoshige and M. Fujita, Fourth Pacific Rim

Int. Conf. on Advanced Materials and Processing (PRICM4), Vol.1, pp.899-902 (2001) The Japan Institute of Metals.
2) J.-S. Lee, K. Hokamoto and K. Siva Kumar, Second Int. Conf. on Light Materials for Transportation Systems (LiMAT-2001), Edited by Nack J. Kim, C. S. Lee and D. Eylon, Vol.II, pp.811-816 (2001) Pohang University of Science and Technology.
3) K. Siva Kumar, P. Soloman Raj, T. Balakrishna and K. Hokamoto, J. Mater. Process. Technol., 115-3, 396 (2001).
4) K. P. Staudhammer and K. A. Johnson, in "Metallurgical Applications of Shock-Wave and High-Strain-Rate Phenomena", Edited by L. E. Murr, K. P. Staudhammer and M. A. Meyers, pp,149-166 (1986) Dekker.
5) S. Itoh, S. Kubota, A. Kira, S. Nagano and M. Fujita, J. Japan Explosives Society, Vol.55, pp.71-94 (1994) (in Japanese).
6) K. Hokamoto, S. Tanaka and M. Fujita, Int. J. Impact Eng., 24, 631 (2000).
7) B. D. Cullity, "Elements of X-Ray Diffraction", pp.284-285 (1978) Addison-Wesley Reading.
8) L. H. Yu and M. A. Meyers, J. Mater. Sci., 26, 601 (1991).
9) M. A. Meyers, N. N. Thadhani and L. H. Yu, in "Shock Wave for Industrial Application", Edited by L. E. Murr, pp.265-334 (1988) Noyes Publications.
10) K. Kondo, in "High Pressure Explosive Processing of Ceramics", Edited by, R. A. Graham and A. B. Sawaoka, pp.227-282 (1987) Trans Tech Publications.