Various microstructures suggesting possible shock compaction mechanisms

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In order to reveal mechanisms of the shock compaction process for metallic powders, a shock recovery experiment for equiatomic NiTi alloy powder was performed using the flyer plate impact technique, and the microstructure at the central region of the sample after the shock treatment was observed under a metallurgical microscope. It is clearly confirmed from the microstructures that the powder is compacted by three mechanisms: (1) generation of a molten metal jet and its trapping, (2) dynamic friction between powder particles, and (3) plastic deformation around a void. The microstructure of the shock-compacted powder depends on the range of particle dimensions, which implies that the compaction process based on the above mechanisms is influenced by the powder particle size and its distribution. At the periphery of the sample, two structural features are observed. One, which is characterized by the large melt pool and the marked deformation and breakage of the powder particle, is located near the sample region to which a plane shock wave propagates through the sample capsule. The other shows the long melt band along the bottom and the side of the sample. These features are attributable to a shock wave with a nonuniform shock front and a radial shock wave, respectively.

1. Introduction

It is known in the shock compaction of metallic powders that the deformation and melting of the particle surface occur during the passage of a shock wave through the powder and lead to void elimination and interparticle bonding [1–10]. Such a phenomenon in shock compaction implies that a relatively large amount of energy generated by the shock loading is heterogeneously produced at the particle surface region because of the strong interaction between powder particles. This unique interaction is important in compacting a powder because an elevated temperature at the surface region makes it easy to deform particles and the melt produced fills initial voids. Until now, in order to explain a microstructure after the shock treatment, phenomenological approaches for the shock energy expansion have been performed in view of the model of the surface melting and the thermal conduction, which are in good agreement with experimental results [6–9]. Some mechanisms of surface heating, such as microplastic deformation, friction between particles, etc., have been suggested in a previous paper [11], and the shock-compacting process of a metallic powder has been simulated on the basis of the plastic deformation mechanism using a two-dimensional finite difference method [12]. However, the shock-compaction process with preferential energy deposition has not yet been satisfactorily elucidated, because the compaction behaviour for a powder may be a complicated construction of some even minute mechanisms such as particle size.

Recently, the shock recovery experiment for the equiatomic NiTi alloy powder has been performed using flyer plate impact technique [13, 14]. That material has the unique property of a shape memory attributed to the thermoelastic martensitic transformation near room temperature. The shock consolidation process of the NiTi alloy powder is intrinsically taken to be the same as for other metallic powders because in a proper shock condition the melt region is obviously observed between the powder particles. As the shock energy increases, it is usually difficult to analogize the mechanisms of heterogeneous expansion of the shock energy by observing the metallurgical structures of the shock recovered samples, because a microstructure rapidly cooled from melt is introduced by the mixing of multiple compaction mechanisms even at a small part of the powder particle size or is translated into a recrystallization structure by a large amount of shock energy.

For a better understanding of the compaction mechanisms by a shock wave, it is helpful to observe a powder compacted by a minor shock energy where the microstructure due to a single mechanism may remain, separated from the compaction behaviour complicated by the mixing of some mechanisms. In

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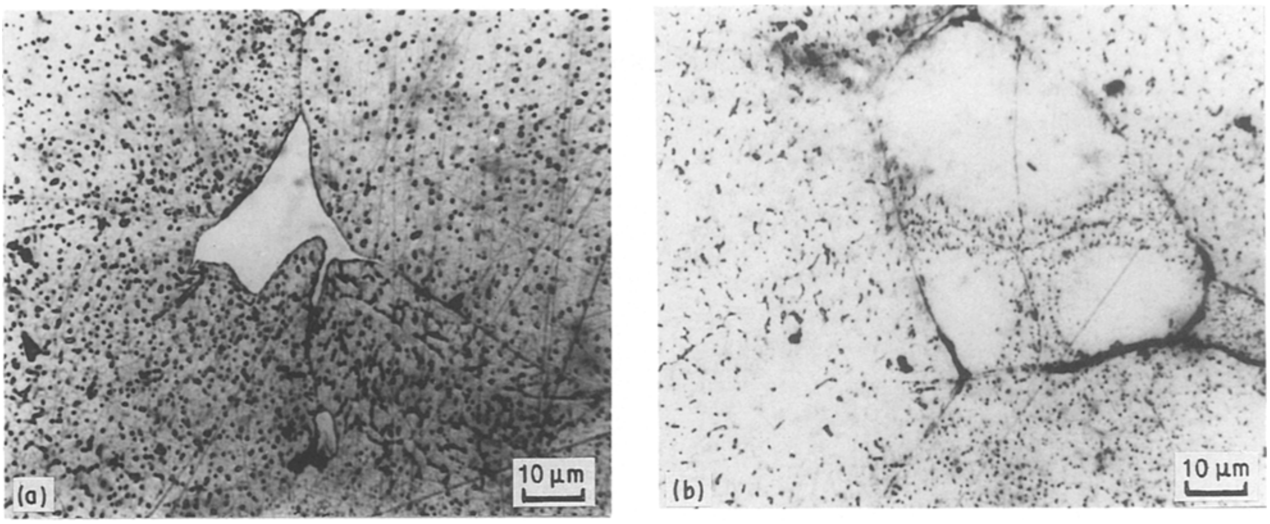

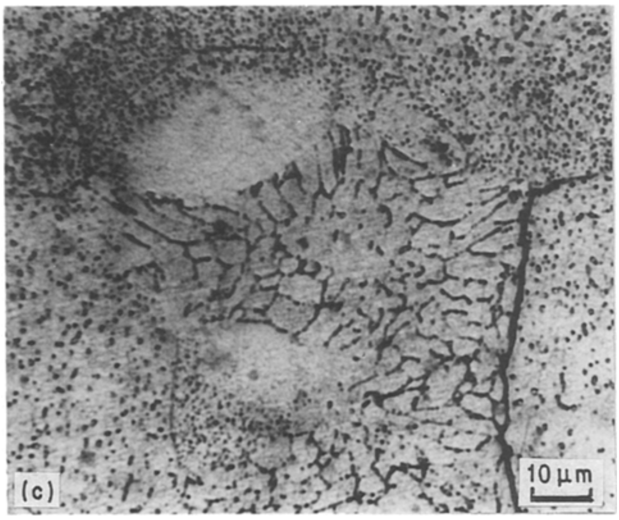

Figure 1 Microstructures of the cross-section of sample N703 due to the compaction mechanisms: (a) jetting and trapping of molten metal, (b) dynamic friction between powder particles, and (c) plastic deformation around a void.

the present paper, the microstructure of the equiatomic NiTi alloy powder after shock treatment at a low flyer velocity, that is, the minor shock energy, was examined in detail under a metallurgical microscope. In addition, an experiment for the NiTi alloy powder with a restricted distribution in the particle diameter was also performed.

2. Experimental procedure

The equiatomic NiTi alloy powder prepared by a centrifugal atomization method was supplied by Daido Special Steel Co Ltd (Minami, Nagoya, Japan). The alloy particles were generally spherical with a wide range of diameters: ~ 50 to $200\,\mu$m. The alloy powder with a restricted diameter range was prepared by sieving. The powder was pressed to a density of 70% full density and then dynamically compacted by the flyer plate impact technique, using a powder gun. The Hugoniot parameters for the present experiment were estimated numerically on the basis of the Rankin–Hugoniot equations, the Mie–Grüneisen equation of state and with some assumptions. Details of the compaction procedure and the numerical estimation are presented elsewhere [13]. The cross-section of the shock-treated sample was polished and then etched in an acid solution of $H_2O:HNO_3:HF = 40:1:3$ for observation under a metallurgical microscope. The microVicker's hardness, if necessary, was measured by loading with a weight of 200 g for 30 sec.

3. Results and discussion

In the present experiment, the flyer velocity, particle size range of the initial powder and the values of Hugoniot parameters for the powder under the shock condition are shown in Table I. Four shots are selected from a series of shots to demonstrate and compare the microstructures introduced by typical compaction mechanisms. Samples are compacted at flyer velocities below the optimum velocity for compaction in the previous paper [13, 14], which have two types of particle diameter distributions: wide and narrow. The microstructures at the central parts which are taken to be compressed to solid density with a plane shock wave, are shown in Figs 1 to 4. In addition to the compaction process with a plane shock wave, two structural features observed near the sample capsule are indicated in Figs 5 to 7.

The microstructures of sample N703 are shown in Fig. 1a to c. This shot is shock-treated at a flyer velocity of $0.65\,\mathrm{km\,sec^{-1}}$, or compacted with small

TABLE I Experimental characteristics

Shot number	Particle diameter range (μm)	Flyer velocity ($\mathrm{km\,sec^{-1}}$)	Shock velocity ($\mathrm{km\,sec^{-1}}$)	Shock pressure (GPa)	Shock energy ($\mathrm{kJ\,kg^{-1}}$)
N703	~ 50 to 200	0.65	1.71	4.2	140
N706	~ 50 to 200	1.31	2.92	12.9	447
N7012	53 to 105	1.38	3.02	14.3	490
N7013	53 to 105	1.04	2.47	9.0	306

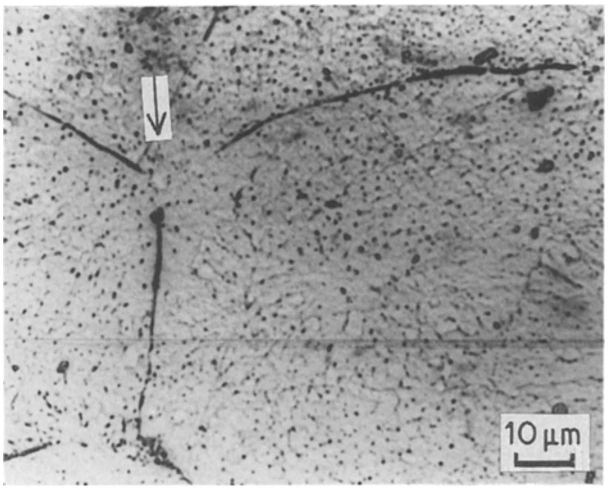

Figure 2 Microstructure of sample N7012. The microstructure is produced by the compaction mechanism of the plastic deformation around a void.

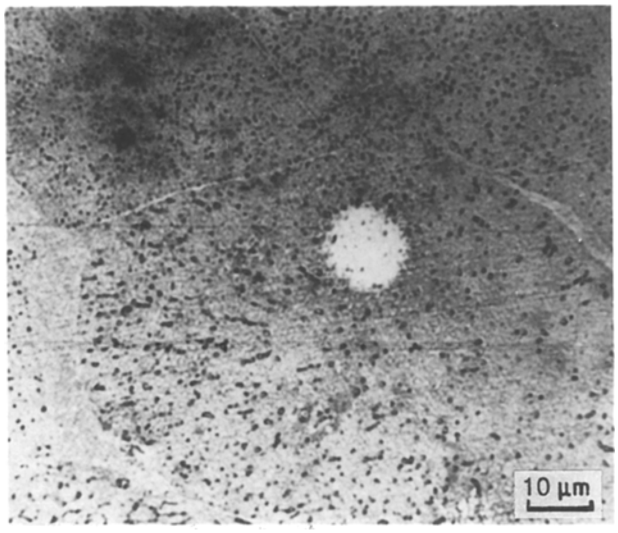

Figure 4 Microstructure of sample N7013. The microstructure shows the melt spot inside a powder particle which is attributable to the generation of a metal jet with a very high temperature.

shock energy. The shock pressure and the shock energy are 4.2 GPa and 140 kJ kg^{-1}, respectively. The melt part, which is surrounded by four powder particles, is shown in Fig. 1a. The melted region is evidently distinguishable from the powder particles because of the presence of the clear boundary, and it is harder by about 50 microVicker's hardness than the surrounding particles. The hardening is considered to be the effect of vacancies caused by rapid cooling. It is clear that the microstructure and hardening show the generation of a molten metal jet and its subsequent quenching. Therefore, it can be speculated that the intergranular bonding between powder particles is achieved by the formation of a metal jet which becomes trapped at the clearance between the particles and is cooled rapidly.

The microstructure of a powder particle surrounded by larger particles is shown in Fig. 1b. This is a unique structure with the melted parts only inside the smaller particle near points of contact of three particles. It is thought that the structure of Fig. 1b may be attributed to a complicated relative motion of the powder particles during the passage of a shock wave, that is, a remarkable dynamic friction.

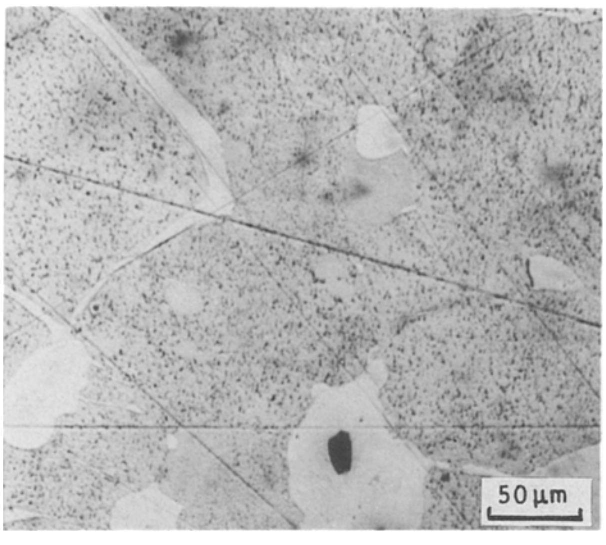

Figure 3 Microstructure of sample N706. The melt region quenched is clearly seen compared to sample N7012 as shown in Fig. 2.

A recrystallized structure, which is enclosed with two small parts rapidly cooled from melt, and the non-recrystallized region, is shown in Fig. 1c. The compaction process for such a microstructure is taken to be different from that mentioned above because most of the area influenced by heat is annealed and has a recrystallization texture in spite of the similar size to the portion quenched from the melt, as shown in Fig. 1b. The microstructure as shown in Fig. 1c is attributed to the mechanism of particle deformation during shock compression, because it is speculated that the region around an initial void is affected by marked plastic deformation with a high stress field, where the energy depending on the strain and the shock pressure during the compaction, is deposited, and the marked deformation region is spread over a wider range than the minute parts concerned with the metal jet and the friction mechanisms. Therefore, the deformation mechanism to crush an interstice between powder particles can not concentrate the shock energy within a very narrow zone, and this results in the recrystallized structure instead of the structure quenched from the molten state. Furthermore, the microstructure of Fig. 1c may accompany not only particle deformation but also dynamic friction between

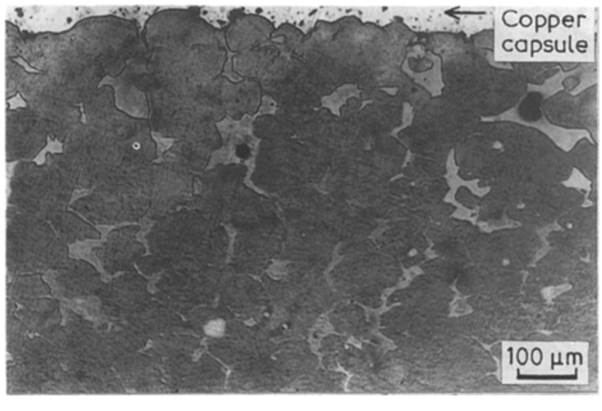

Figure 5 Microstructure of sample N7013. A larger melt pool in the upper part is formed in comparison with the lower part.

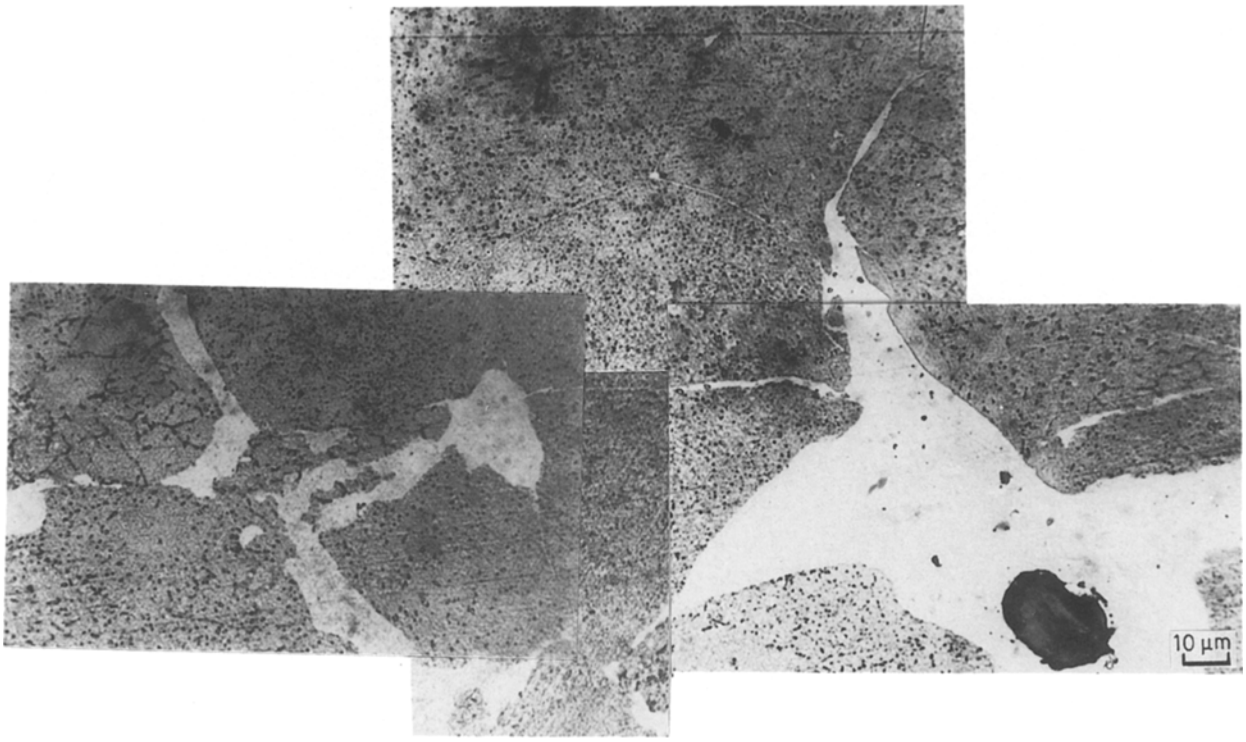

Figure 6 The microstructure of the upper sample region of N7013. The powder particles are markedly deformed and broken.

particles because of the wide distribution in particle diameter. The typical structure of the sample with a narrow diameter distribution is indicated below.

The microstructure near a point of contact of three particles with a similar size is shown in Fig. 2. Such a structure can often be seen in sample N7012. The intergranular bond is sufficiently strong, though the melting structure is not necessarily apparent. It is speculated that the bond will result mainly from pressure welding and heating due to the deformation. On the other hand, the particle boundaries disappear near a point of contact of three particles as indicated by the arrow. The formation of such a microstructure is attributable to a relatively large heat generation due to the large deformation when the interstice between particles is crushed under the shock pressure. Therefore, it is clear that the microstructure in Fig. 2 shows the mechanism of a shock compaction process due to mechanical deformation around a void. This microstructure is in qualitative agreement with the results simulated by Williamson and Berry [12], where the final particle shape and thermal history on the shock compaction for the 304 stainless steel particles with a diameter of 75 μm is shown on the basis of the deformation to crush voids. In Fig. 2, the microstructure is homogeneous without a clear melt structure, which is completely unlike the microstructures due to the mechanisms of metal jet creation and trapping, and friction, as shown in Figs 1a and b. Obviously, this suggests that the part heated by particle deformation is wide in comparison with those due to the other two mechanisms, though the quantity and the range of the heat evolution in a minute part depend on the particle size and the shock conditions.

In the case of sample N706, as shown in Fig. 3, the melt zone is readily distinguishable from other parts which behave as a heat sink for quenching, and the microstructure is very different from that in sample N7012, as shown in Fig. 2, in spite of the similar shock conditions. This implies that the microstructure of the shock-loaded powder is remarkably influenced by the range of powder particle size, and the shock energy produced by the process of the particle deformation decreases relatively with increasing distribution range of particle diameter.

The melt spot inside a powder particle for sample N7013 is shown in Fig. 4. A terrible metal jet impinges on the powder particle and melts a minute transgranular region. Therefore, the metal jet has a very high temperature in comparison with one in the compacting condition of the low shock energy as shown in Fig. 1a. However, the transglanular melt area is very small and hardly appears even under high shock energy conditions.

For the shock compaction of the powder, a metallic capsule for use as a sample holder is necessarily to prevent dispersion of the powder sample during shock treatment. Therefore, it is important in shock processing of the powder to elucidate the influence of the sample capsule on the microstructure of the shock-loaded sample. Two unique structures of the compacted sample can be pointed out as effects due to the sample capsule. One of the structures, as shown in Figs 5 and 6, is located in the upper sample region to which a plane shock wave propagates through the sample capsule, while another one, as shown in Fig. 7, is seen in the long band region along the bottom from the upper corner of the compacted sample. The upper region of sample N7013 shows the characteristic microstructure as shown in Figs 5 and 6. Fig. 5 shows that the melt part is large in comparison with that of the lower or centre region of the sample. Fig. 6 shows the remarkable deformation and breakage of the powder particle. It is speculated from these micro-

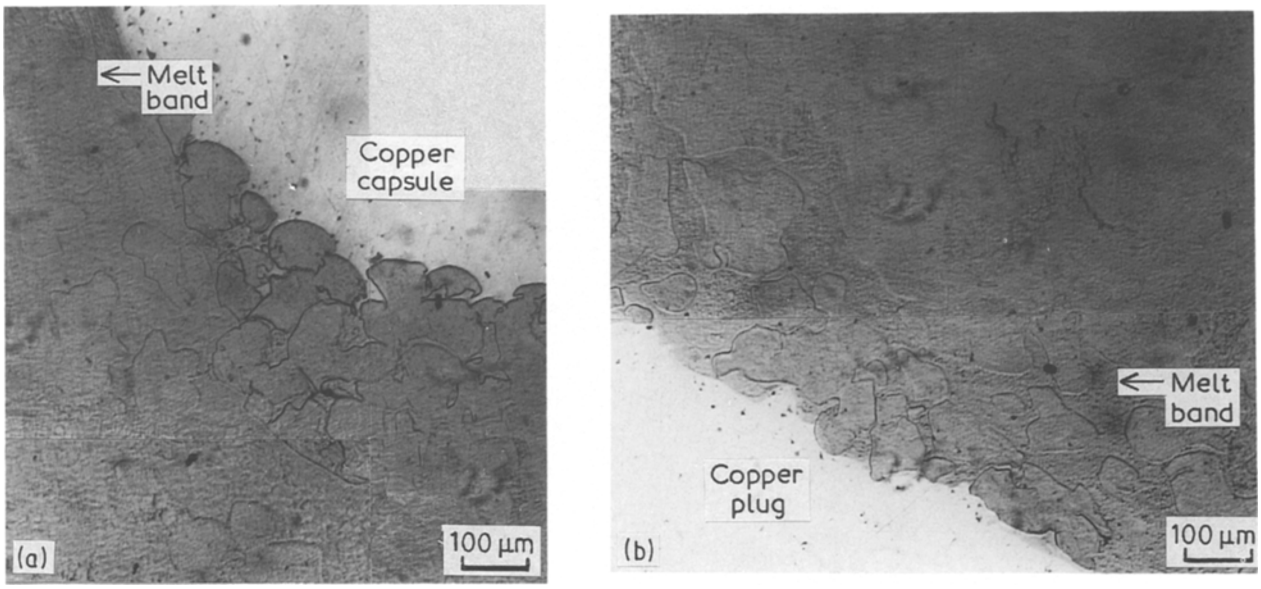

Figure 7 Microstructures of sample N7012 showing the long melt band in the peripheral part from the (a) upper corner to (b) the bottom of the sample.

structures that the upper region is shock compacted by an unsteady shock wave, that is, a shock wave with a nonuniform shock front, where shock-loaded and unloaded particles are transiently generated and the marked particle deformation and particle breakage due to the spalling phenomenon for the particle occur, in addition to the formation of the large melt part between the particles by the dynamic friction and the metal jetting mechanisms. As the shock wave propagates in the powder, the shock front becomes more uniform, and the central region of the sample shows a more uniform microstructure with a layered surface melt and a small deformation of the powder particle as shown in the lower region in Fig. 5. Therefore, it is clear that such a unique structure shows the transient state of the shock compaction due to the nonuniformity of the shock front when the shock wave propagates from the sample capsule to the powder, and if a shock wave with an inhomogeneous shock front can be advanced through a long distance in the powder by considering the shape of the interface between the capsule and the powder sample, arrangement of the powder particles, etc., a material with a unique microstructure may be formed by more heterogeneous production of the shock energy.

On the other hand, a long melt zone is formed along the bottom from the upper corner of the sample. The peculiar structure is shown in Figs 7a and b which are from near the upper corner and the bottom of sample N7012, respectively. In addition to the plane shock wave from the upper side of the sample, an additional shock wave enters the powder from the side of the sample, because the shock wave in the sample capsule made of copper has a rapid shock velocity and a high shock pressure compared to those in the powder, and the additional wave propagates as a radial shock wave through the powder. Therefore, the long melt zone is considered to be the very compacted region where the plane shock wave collides with the radial shock wave. This melt band is clearly seen in the sample compacted over velocities of about 1 km sec^{-1}, which implies that the shock energy produced in this zone is more than double that in the other region compacted with only the plane shock wave. Although such an effect of the capsule must be removed to make a homogeneous microstructure, it is helpful in synthesizing compounds of materials which are difficult to react and for treating samples under more stringent shock conditions.

Based on observations of the microstructure of the shock-loaded NiTi alloy powder, it is clearly confirmed that the metallic powder is compacted by three mechanisms: a metal jet, dynamic friction and plastic deformation. Although the question of what happens in the region of atomic size cannot be answered from the present results, for a more intrinsic understanding of the shock compaction these mechanisms will have to be explained by the process in the smaller regions, such as the high-speed movement and multiplication of dislocations, the interaction between atoms, etc. The dependence of the microstructure upon the particle size distribution is considered to be helpful for a better comprehension of powder compaction which clarifies the difference between the shock compaction mechanisms. Furthermore, it is important in the application of shock compaction to take not only the shock conditions but also the range of particle diameters into account in order to control the microstructure.

The structural features as shown in Figs 5 and 6 are based on the intrinsic powder compaction process related to the propagation behaviour of the shock wave in the solid and powder materials, and another feature, as shown in Fig. 7, is produced by the recovery assembly, depending on the material and the size of the sample capsule. These features were seen in the every sample compacted at the appropriate flyer velocities from about 1.0 to 1.4 km sec^{-1} in order to observe the melt structure. The effect of using the sample capsule as described above must be satisfactorily considered in an analysis of the microstructure of the shock-compacted powder sample.

Therefore, the inhomogeneous microstructure formed by shock compaction is attributed to the compaction mechanisms and the effect of the sample capsule, and for the purpose of making a more homogeneous compact, it is necessary to consider the particle shape, the distribution of the particle size and the factors of the sample capsule such as the material, the shape and the size, in addition to the shock pressure and the shock energy.

4. Conclusions

Shock compaction for the NiTi powder sample was carried out under conditions of low shock energy, and that for the powder with a restricted particle diameter range was also performed using a powder gun. The microstructures in the central region of the recovered samples were observed, and the typical shock compaction mechanisms were revealed as follows:

1. generation of a molten metal jet and its trapping;
2. dynamic friction between powder particles;
3. plastic deformation around a void.

Although the microstructure due to one of the compaction mechanisms is quenched in a minute part under shock conditions of low shock energy, compaction under ordinary shock conditions is usually made through the complicated process where above three mechanisms are combined. The mechanism of the generation of a metal jet is able to heat the jet to a very high temperature which melts a minute transgranular portion of the neighbouring particle. The dynamic friction tends to become a mechanism of shock compaction between large and small particles. The microstructure for the plastic deformation mechanism is often seen in the compacted powder with a narrow particle diameter distribution. The deformation area around a void, where the shock energy is produced, is wide compared with regions affected by the other two mechanisms, because the clear melt structure is not seen.

Two influences due to the use of the sample capsule to prevent dispersion of the powder during shock loading are observed in the microstructure near the capsule. One is located in the upper region of the compacted sample, where the microstructure is characterized by the large melt pool, and the remarkable deformation and breakage of the powder particle. It is speculated that this region shows the transient compaction state with a nonuniform shock front when the shock wave propagates from the capsule to the powder. The other influence is shown by the long melt band along the bottom from the upper corner of the sample where the plane shock wave collides with the radial shock wave due to the differences in the shock impedance between the capsule and the powder.

The microstructure of the shock-compacted powder is very inhomogeneous because of the compaction mechanisms and the influence of the sample capsule, which often combine and lead to the complicated compaction process.

Acknowledgements

We thank Professor A. Sawaoka of Tokyo Institute of Technology for supporting the shock experiment and Dr K. Kusaka of Daido Special Steel Co. Ltd for supplying the powder.

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Received 11 August 1988
and accepted 11 January 1989