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Powder Consolidation by Severe Plastic Deformation and its Applications in Processing Ultrafine and Nanostructured Aluminium and Composites

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Severe plastic deformation (SPD) based processes have shown great potential in producing bulk ultrafine and nanostructured materials. In addition to refining grains, SPD processes such as equal channel angular pressing (ECAP) and high pressure torsion (HPT) can be used to fabricate bulk materials from particles. It enables particles from nano to micro scales to be consolidated into fully dense materials at much lower temperatures and shorter times, compared to conventional sintering. It is especially suitable for producing complex multiphase alloys and nanocomposites. In this paper, the mechanism of SPD consolidation is illustrated using the example of equal channel angular pressing (ECAP) of micro- and nano-scaled pure Al particles. It is shown that consolidation and good bonding require the particles to undergo plastic deformation rather than sliding over each other. In addition to monolithic materials, Al-C composites have been produced in combination with mechanical milling. In another example, Al and Ti particles have been mixed and consolidated using a combination of mechanical alloying and ECAP to obtain ultrafine and nanostructured alloys. In all cases, the mechanical behaviour is dependent on the scale and distribution of the phases, and both strength and ductility can be significantly improved by finer sizes and more uniform distribution, which are achievable through SPD processing.

Keywords: *Severe plastic deformation; equal channel angular pressing; powder consolidation; ultrafine; nanostructured.*

1. Introduction

Ultrafine and nanostructured metals and alloys and metal matrix nanocomposites (MMnCs) have attracted considerable research efforts for their promised properties such as simultaneously improved strength and ductility and enhanced superplasticity [1, 2]. These materials can either be built up by assembling ultrafine/nano particles in the so-called bottom-up approach, or be produced by refining an originally coarse microstructure (top-down). Among the latter, severe plastic deformation (SPD) has turned out to be most effective [3]. In particular, equal channel angular pressing (ECAP) [4] and high pressure torsion (HPT) [5] are the two most employed processes. ECAP has the capability of making large-size bulk material with uniform grain structures whereas HPT can impose considerably higher strain at lower temperatures although the sample sizes are quite limited.

However, there are limitations in these two approaches. Assembling nanoparticles is often a slow process and can only produce relatively small bulk material most likely with residual pores. It might require high temperatures which would have destroyed any nanostructures existing in the particles. On the other hand, the grain sizes achievable through SPD is determined by the balance between refinement and coarsening, with the finest grains obtained to be several hundreds of nanometres [6]. It is therefore of interest to introduce a third approach which combines the bottom-up and top-down ones to produce bulk ultrafine and nanostructured materials by using SPD to consolidate fine particles [7]. Promising results have been obtained in some common metals and alloys including Al, Cu, Ti, Mg and Ni [8-19].

Consolidation of particles through SPD differs from the conventional sintering process in that it relies on plastic deformation of the particles, rather than diffusion, to achieve good bonding and full density [20]. It offers advantages of processing at considerably lower temperatures and much shorter processing times with resulting materials free of residual pores and other defects associated with conventional sintering. Consequently, bulk materials of high integrity can be made from particles with special structures and compositions including nanocrystalline, amorphous and high alloy contents produced by such non-equilibrium processing as mechanical alloying and rapid solidification. It is also most suitable for making nanostructured composites since different phases of various sizes can be easily combined. In this paper, the mechanism of SPD consolidation will be briefly described and a variety of ultrafine and nanostructured Al based materials and composites processed by SPD consolidation will be introduced.

2. Mechanism of SPD Consolidation [20]

Metal particles do not bond to each other spontaneously owing to the existence of surface oxide layers. In the conventional sintering process, the surface layer is broken down by diffusion which requires long time at high temperatures. The lack of material flow other than that caused by diffusion also results in residual pores in many cases, and the prior-particle boundaries (PPBs) are mostly not disrupted. The use of high temperatures not only demands greater energy consumption, but more importantly, causes any fine (especially ultrafine and nano) and non-equilibrium structures which might exist in particles to disappear. In the case of nanocomposites, the nanoparticles are often clustered, causing significant loss of any advantages.

In the case of consolidation by SPD, the particles are forced to undergo shape change by the plastic deformation process used (obviously the particles amenable to SPD consolidation have to be deformable, that is, not brittle). Since the surface oxide shell cannot deform plastically, it will be ruptured, leaving freshly exposed metal surface to be in direct contact with each other. Consequently, bonding will take place spontaneously without needing diffusion. Moreover, material flow would take place in SPD consolidation and this leads to the filling of any gap between particles and thus elimination of pores. The process is illustrated in Fig. 1 with comparison to conventional sintering.

It is also obvious from Fig. 1 that the original oxide surface layer (the black shell in Fig. 1) would be broken into pieces (often of nano-sizes) which are distributed in the matrix by the plastic deformation process (second line in the right column in Fig. 1). In the case of ECAP, the distribution can be made more uniform with increasing number of passes. This would completely remove PPBs, and potentially can turn these nanoscaled particles into strengthening elements.

It is critical to achieve complete consolidation and good bonding that the particles actually be deformed rather than slide over each other, as demonstrated in examples in [20]. That

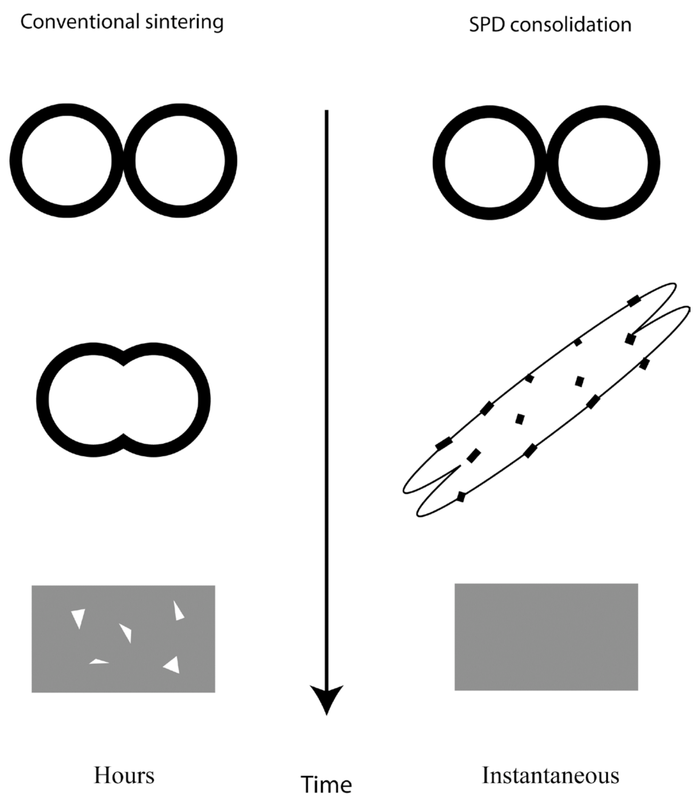

Fig. 1 Comparison of the conventional sintering (left) and SPD consolidation (right) processes [20].

is, the strength of the particles should be less than the resistance to sliding between particles. Softer, larger and irregular-shaped particles are thus easier to consolidate. However, the particles desirable for high performance are often strong and small (e.g. nano-sized or nanostructured), and processing parameters might need to be adjusted to be able to consolidate them well; in the case of ECAP, this might involve increased back pressure and higher temperature if allowed.

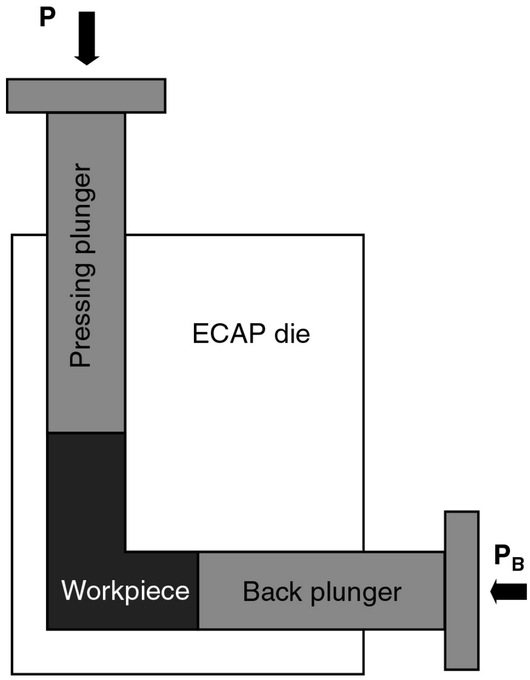

Fig. 2 Illustration of the back pressure equal channel angular pressing process used [10].

3. Aluminium Based Materials Processed by SPD Consolidation

Pure aluminium is soft and ductile, and ideal for SPD consolidation. In the following sections, examples of Al and Al matrix composites produced by SPD consolidation are presented to demonstrate the effectiveness and versatility of the method. In all the cases, the process used is back pressure equal channel angular pressing (BP-ECAP) as illustrated in Fig. 2. The difference from the standard ECAP is that a constant back pressure is applied through the back plunger in the exit channel. This is desirable and even essential in SPD consolidation to provide constraint and more importantly to prevent particle sliding [20].

3.1 Pure Al from micro-sized particles

The easiest case is when micro-sized pure Al particles of irregular shapes are used [10, 11]. Full consolidation can be achieved after only one pass at as low as room temperature with a moderate back pressure of 50 MPa. After four passes at 100°C following Route B_c (i.e. rotating samples by 90° in a single direction between passes), the grains were refined to < 1 μm and yield strength increased to 170 MPa.

This is in comparison to a sintering temperature of around 600°C with a much coarser grain structure and lower strength in conventional powder metallurgy processing. Therefore, SPD consolidation can be used in compacting and consolidating Al particles with potentially considerable cost saving and better properties.

3.2 In-situ nanostructured Al-Al_2O_3 composites from nano-sized particles

The situation was very different when the pure Al particles used had an average size of ~100 nm (compared to 34 μm for micro-sized particles) and a spherical shape. These made the nano particles much stronger and more susceptible to sliding. Indeed, full consolidation was not achieved after one pass at a much higher temperature of 400°C and back pressure of 200 MPa because most particles were not deformed [12]. The particles were forced to shear deform in subsequent passes, leading to full consolidation and a nanocrystalline structure of Al and Al_2O_3, shown in Fig. 3. Consequently, the strength reached 740 MPa although the material was brittle owing to the high alumina content and its non-uniform distribution.

The significant amount (~30 vol%) of alumina in this material was formed in-situ through continuous oxidation during ECAP which was performed in air (increased from < 5 vol% in the as-received particles and ~18 vol% after 2 passes) [13]. The interaction of oxide formation and shear deformation gave rise to the nanocrystalline structure observed in Fig. 3. This demonstrates the possibility of using ECAP consolidation to produce in-situ MMnCs (i.e. without adding alumina particles). By controlling the process, the amount and distribution of the oxide particles may be optimised to give the best combination of strength and ductility.

3.3 Al-C nanocomposites

More conventionally, MMnCs can be produced by incorporating nanoscaled particles to Al ones. Nanoparticles, however, easily form agglomerates which are very difficult to disperse [21]. By combining mechanical milling and SPD consolidation, uniform distribution of nanoparticles can be achieved.

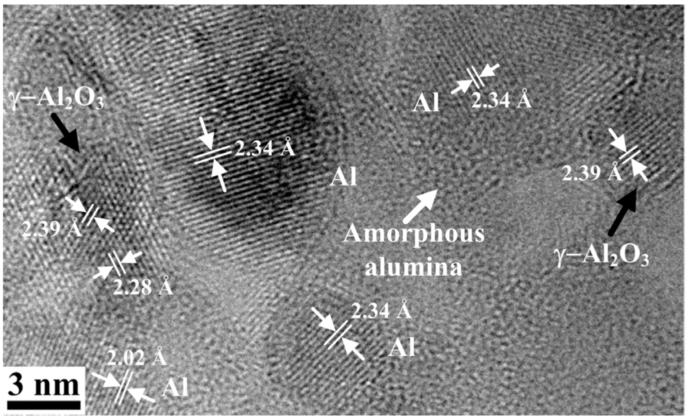

Fig. 3 Nanocrystalline Al and Al_2O_3 in a material consolidated from nano Al particles by BP-ECAP at 400°C for four passes [12].

In the present example, carbon black (CB) was used as the source of carbon nanoparticles. CB is much cheaper than the more commonly used nanoparticles such as SiC. However, the clusters formed in CB are more difficult to disperse. In our investigation [22], ball milling was used first to break the agglomerates which could be as large as 500 nm in size and to disperse individual C particles (~30 nm on average) in Al. The milled mixture was then consolidated by BP-ECAP at 400°C with a back pressure of 200 MPa. TEM observations revealed that the individual C nanoparticles were dispersed in the Al matrix, as shown in Fig. 4 for a Al-5 wt% C material. The presence of the C nanoparticles also helped the refinement of the matrix Al grains to < 500 nm. These together have increased the strength of the composites to the order of 300 MPa from that of 100 MPa in the unreinforced Al.

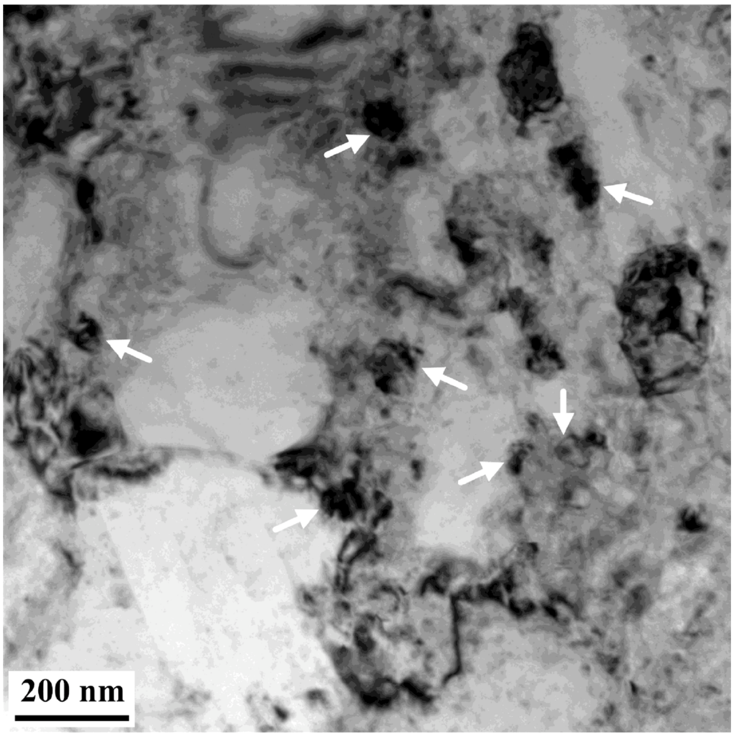

Fig. 4 Individual nano C particles (arrowed) distributed in the Al matrix following mechanical milling of the mixed powder and ECAP consolidation at 400°C for 8 passes [22].

3.4 Al-Ti dual phase materials

SPD consolidation is particularly suitable for multiphase materials, including mixed particles of different sizes [23]. For instance, a dual phase Al-Ti material was produced by consolidating a mixture of micro-sized elemental Al (average size of 34 μm, 47 at%)) and Ti (96 μm) particles. To avoid the formation of intermetallic phases between Al and Ti, the consolidation temperature used was 350°C. In one case, the ECAP consolidation was done on the mixed powder directly, resulting in coarse Ti particles in the matrix of Al. In the other case, the mixed powder was mechanically milled first before consolidation, leading to micro-scaled Ti grains as well as nano-sized Ti particles in the matrix of nano-scaled Al grains.

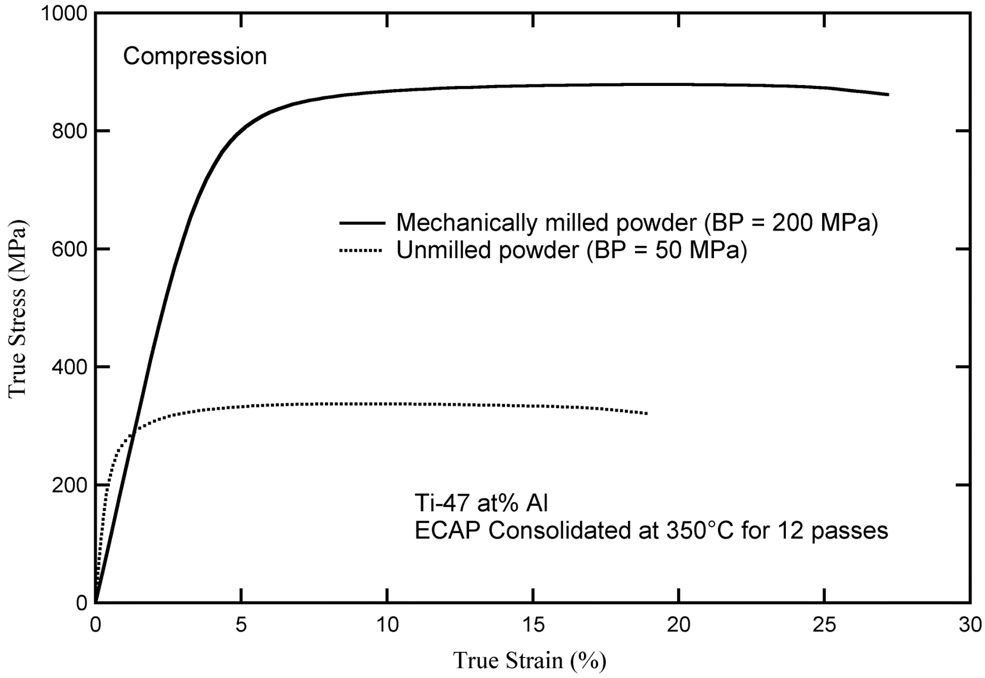

Fig. 5 Compressive curves for two Ti-47 at% Al materials consolidated by BP-ECAP at 350°C.

Figure 5 shows the compressive curves for the two materials after BP-ECAP consolidation at 350°C for 12 passes. It is apparent that the one from the milled powder was much stronger with a yield strength of ~800 MPa, compared to ~250 MPa for the unmilled powder. This is attributed to the much finer microstructure in the former.

4. Summary

It has been demonstrated that SPD consolidation (BP-ECAP in particular) is effective and versatile in producing bulk ultrafine and nanostructured Al and Al composites from a variety of particles. It relies on plastic deformation rather than diffusion to achieve good bonding and full density and thus can be carried out at considerably lower temperatures and shorter times compared to conventional sintering. Single phase Al, Al matrix nanocomposites and multiphase Al based materials have been processed with significantly enhanced strength. Potential applications may range from simply compacting and consolidating powders (cost saving and high quality) to processing materials with designed microstructures and properties.

Acknowledgements

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References

1. H. Gleiter: Acta Mater. 48 (2000) 1-29.
2. C. C. Koch: J. Mater. Sci. 42 (2007) 1403-1414.

3. R. Z. Valiev: Nature Mater. 3 (2004) 511-516.
4. R. Z. Valiev, T. G. Langdon: Prog. Mater. Sci. 51 (2006) 881-981.
5. A. P. Zhilyaev, T. G. Langdon: Prog. Mater. Sci. 53 (2008) 893-979.
6. R. Pippan, F. Wetscher, M. Hafok, A. Vorhauer, I. Sabirov: Adv. Eng. Mater. 8 (2006) 1046-1056.
7. K. Xia: Mater. Sci. Forum 579 (2008) 61-74.
8. K. Matsuki, T. Aida, T. Takeuchi, J. Kusui, K. Yokoe: Acta Mater. 48 (2000) 2625-2632.
9. Z. Lee, F. Zhou, R. Z. Valiev, E. J. Lavernia, S. R. Nutt: Scr. Mater. 51 (2004) 209-214.
10. K. Xia, X. Wu: Scr. Mater. 53 (2005) 1225-1229.
11. K. Xia, X. Wu, T. Honma, S. P. Ringer: J. Mater. Sci. 42 (2007) 1551-1560.
12. W. Xu, T. Honma, X. Wu, S. P. Ringer, K. Xia: Appl. Phys. Lett. 91 (2007) 031901.
13. W. Xu, X. Wu, T. Honma, S. P. Ringer, K. Xia: Acta Mater. 57 (2009) 4321-4330.
14. M. Haouaoui, I. Karaman, H. J. Maier, K. T. Hartwig: Metall. Mater. Trans. A 35A (2004) 2935-2949.
15. V. V. Stolyarov, Y. T. Zhu, T. C. Lowe, R. K. Islamgaliev, R. Z. Valiev: Mater. Sci. Eng. A282 (2000) 78-85.
16. W. Xu, X. Wu, D. Sadedin, G. Wellwood, K. Xia: Appl. Phys. Lett. 92 (2008) 011924.
17. X. Wu, W. Xu, M. Kubota, K. Xia: Mater. Sci. Forum 584-586 (2008) 114-118.
18. R. Z. Valiev, R. S. Mishral, J. Grozal, A. K. Mukherjee: Scr. Mater. 34 (1996) 1443-1448.
19. A. Bachmaier, A. Hohenwarter, R. Pippan: Scr. Mater. 61 (2009) 1016-1019.
20. K. Xia: Adv. Eng. Mater. (2010) in press.
21. S. C. Tjong: Adv. Eng. Mater. 9 (2007) 639-652.
22. S. Goussous, W. Xu, X. Wu, K. Xia: Comp. Sci. Technol. 69 (2009) 1997-2001.
23. X. Wu, W. Xu, K. Xia: Mater. Sci. Eng. A493 (2008) 241-245.