JOURNAL OF MATERIALS SCIENCE 21 (1986) 1111–1117

Microstructural changes taking place during dynamic compaction of aluminium powders

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The dynamic compaction of powders is characterized by a complex sequence of pressure loading and shear deformation, adiabatic temperature rise and structural annealing. The relative effects of these factors have been examined by studying the hardness and microstructure of compacted aluminium samples. Particle interiors are considerably shock-hardened, and then partially softened by dislocation recovery. The recovered structures appear to be relatively stable against further softening and recrystallization. The melted zones which occur as pore-filling between powder particles takes place are extremely hard and thermally stable. These zones possess a fine-grained structure after rapid resolidification, contain an oxide dispersion from the oxide films on the original powder surfaces, and are hardened by microvoids from the shrinkage occurring during solidification. The final material structures and properties are therefore variable throughout a given compacted samples, and depend sensitively on many aspects of the material, the powder and the consolidation conditions.

1. Introduction

Dynamic compaction is being extensively evaluated as a means of consolidating powders, particularly of hard or metastable materials where problems are encountered when consolidating by more conventional processes. During dynamic, or shock wave, processing a large number of pressure, shear and temperature changes occur and this form of consolidation can be regarded as a complex means of thermo-mechanical working. Thus shock hardening may arise, as encountered during shock-wave working of fully-dense material, or significant heating and consequent chemical or structural changes can occur. The changes expected within a dynamically-compacted powder material may be even more complex because of the variation of process parameters, particularly the extent of shear concentration and the consequent temperature rise, throughout the material.

Many powders possess significant surface layers having a different composition from the bulk, for example as a result of oxidation during or after atomization. The behaviour of these surface layers during and after consolidation, and the influence of the internally-trapped oxides on the eventual mechanical properties of the consolidated material, are not well understood.

The structures and properties of several dynamically-compacted aluminium powders have been examined on as-compacted material and after heat treatment, in order to evaluate the various influences of dynamic compaction on mechanical properties and the thermal stabilities of the structures which form.

2. Experimental techniques

The material used for study was a commercially-pure (99%) aluminium powder prepared by air-atomization. The particles were irregularly shaped with a mean size of about 150 μm, and clearly possessed a relatively thick oxide layer after atomization. Powders were pressed to a density of 60% full density and then dynamically compacted by the impact of nylon pistons accelerated to velocities in the range 600 to 1800 m sec^{-1} by means of gas-gun launchers. Details of the consolidation procedure have been given previously [1]. By assuming consolidation to full density, and using standard Hugoniot data for the piston material, the impact pressures and energy inputs are deduced by the impedance matching procedure. For the impact velocity range studied, the pressures generated were in the range below 1 to 4 GPa, and the mean temperature rise in the range about 80 to 530° C.

In addition, for subsequent comparison of dislocation structures, a number of solid aluminium impacts were performed, by impacting aluminium flyer plates on to solid, annealed aluminium samples. Impact pressures in the range 2 to 12.8 GPa and pressure pulse durations of 0.34 to 5.9 μsec were produced in this way.

Samples of dynamically-compacted material were annealed for 1 h at temperatures in the range 150 to 600° C. Sections through samples were prepared for microhardness measurements and for optical microscopy. In addition, the fine-scale structures of dynamically-compacted and solid-impacted materials were examined by transmission electron microscopy. Finally, the thermal stability of fine-scale microstructure was examined by *in situ* heating within the transmission microscope.

3. Results

3.1. General microstructure and microhardness

Typical optical microstructures through compacted powder samples after compaction at 0.6 and 2 GPa are

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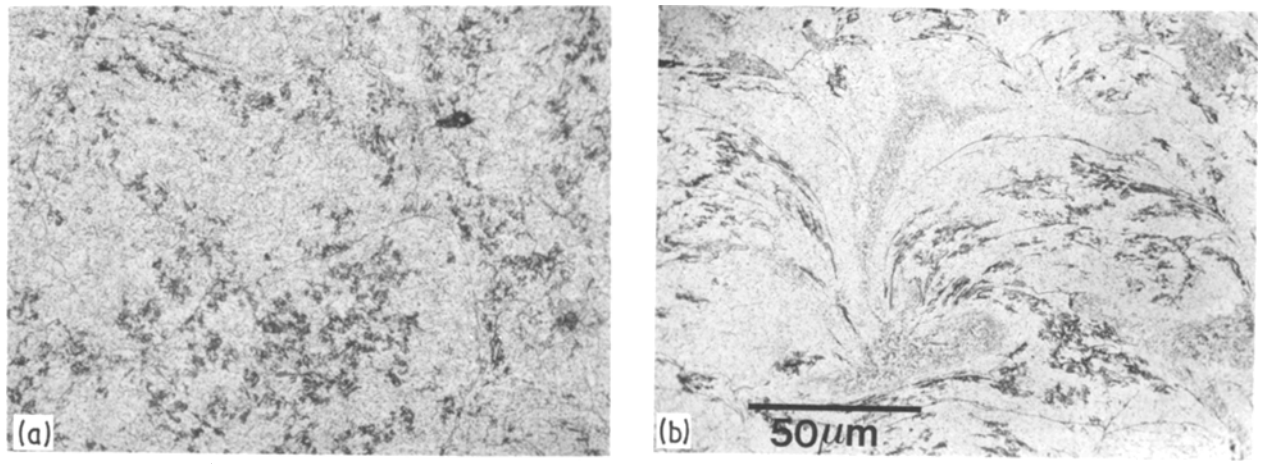


Figure 1 Optical micrographs showing structure of dynamically compacted aluminium: (a) 0.6 GPa, (b) 2 GPa. Shock-wave travel is downwards in both cases. After low-pressure consolidation the particles remain equiaxed with little sign of localized shear concentration or melting. After high-pressure consolidation the particles are flattened in the direction of shock propagation, and extensive deformation and melting occurs as pore-filling occurs.

shown in Fig. 1. After lower-pressure compaction the sample density is already very high, but the prior-particle surfaces are clearly resolvable and the extent of interparticle bonding is minimal. The particles possess flattened surfaces and remain approximately equiaxed with no sign of extensive, localized deformation or melting. After high-pressure consolidation, the particle is considerably flattened in the direction of shock-wave motion as pore-filling takes place [2–5]. Melting has been identified to occur within these extensively deformed regions [3]. Many prior-particle boundaries remain clearly visible even after such high-pressure consolidation, particularly those boundaries lying perpendicular to the direction of shock-wave travel. The anisotropy of bonding which arises leads to anisotropy of toughness in the compacted material [5].

Microhardness results obtained on a dynamically-compacted sample, and also on a cold-worked, solid aluminium sample (commercially pure aluminium, cold-rolled), are shown in Fig. 2 as a function of the annealing temperature used. Measurements were made on the dynamically-compacted material within the massive melted zones, at the prior-particle boundaries (but where no melting was evident), and within the particle interiors. The cold-worked reference material shows a gradual softening up to a temperature of 250° C, and then completely softens, presumably as recrystallization occurs. The dynamically-compacted particle interiors and unmelted boundaries are similar to each other in hardness, with the boundaries being consistantly a little harder, but they differ significantly in annealing response to the cold-worked material. The as-compacted hardness is slightly less than the values of the cold-worked material, but this hardness is maintained or even slightly increased on annealing in the range 150 to 250° C. Thereafter the hardness decreases continuously, but complete softening is not obtained until annealing temperatures about 600° C are used. This retention of strength until high temperature is analogous to that described by Raybould [6] on dynamically-compacted materials. The melted zones are very much harder than any other part of the material, and even though softening begins after annealing at 150 to 250° C, the material remains very hard even after annealing at 600° C. Optical microscopy of these areas showed that some structural coarsening had occurred but that no major recrystallization took place.

Similar variations in microhardness are seen after dynamic compaction at other compaction pressures

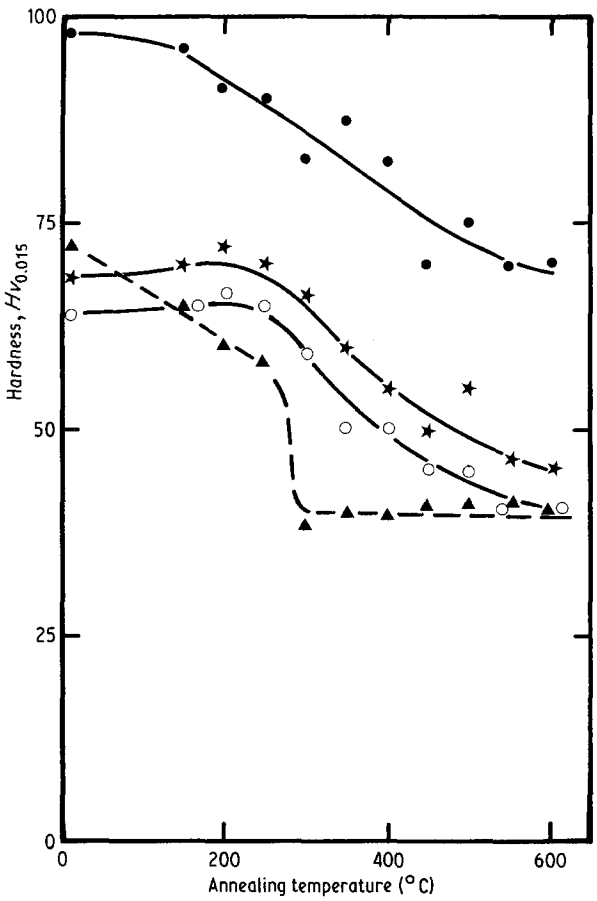


Figure 2 Microhardness on a dynamically compacted aluminium powder sample (○, ★, ●) and on cold-worked, solid aluminium (▲) after annealing for 1 h at each temperature. The interiors of powder particles (○), particle boundaries where no obvious melting occurred (★), and large melted zones (●) within the dynamically-compacted samples have been examined.

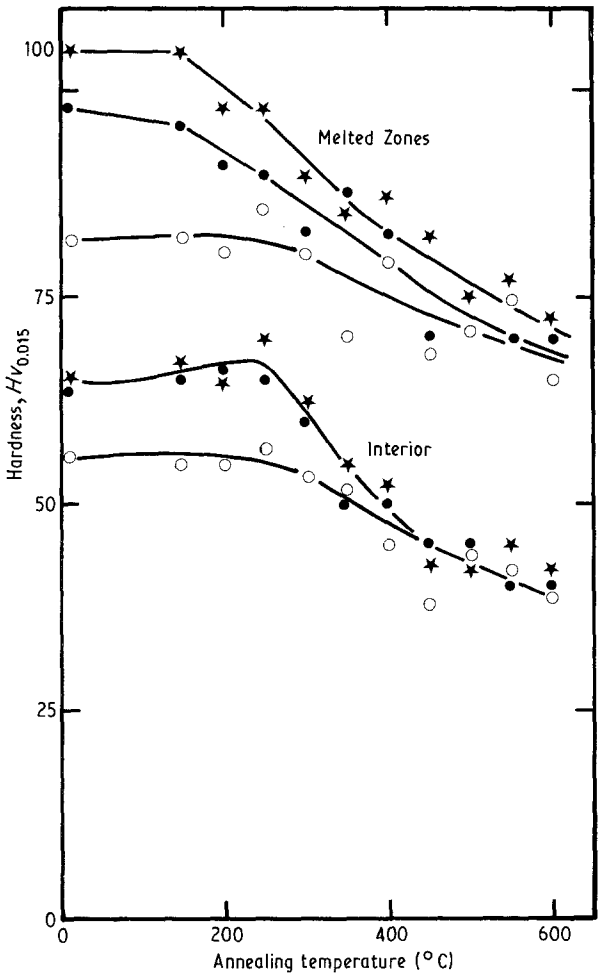


Figure 3 Microhardness on dynamically compacted aluminium powder samples prepared using shock pressures of (★) 2 GPa, (●) 3 GPa and (○) 4 GPa, after annealing for 1 h at each temperature. Hardness values within particle interiors and within melted zones are shown.

(temperatures), see Fig. 3: the hardness is high in all cases, especially at the melted zones; softening takes place gradually such that at 600° C the hardness has finally relaxed (for particle interiors) or is still very high (for melted zones). The hardness of particle interiors shows a slight but noticeable increase when low annealing temperatures are used, but only for compacting pressures of 2 and 3 GPa. It is interesting to note that the hardness achieved, both at particle interiors as well as at melted zones (in fact also at unmelted particle boundaries) varies inversely with the pressure used. Thus the highest pressure, 4 GPa, gave rise, consistantly, to lower hardnesses than the other compacting pressures.

3.2. Transmission electron microscope results

The following micrographs illustrate the detailed structures observed at interparticle boundaries, within melted zones, and within particle interiors. The results reported here supplement those shown previously [3] where interparticle melted structures were emphasized.

Fig. 4 shows part of the junction between two powder particles, where a small amount of melting has occurred, and a region of extremely fine grain-sized material remains. Within such regions, isolated volumes of weakly-diffracting material, apparently oxide,

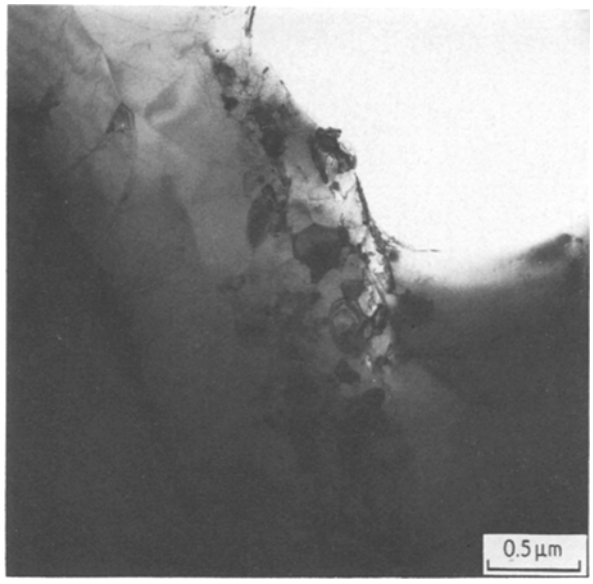


Figure 4 Interface between two aluminium particles in a dynamically compacted sample showing a region of fine grains where melting occurred.

were seen (Fig. 5). The interface between the non-melted particle and the melted region is sometimes clearly indicated by an oxide film. (This figure was taken from an Al–Cu dynamically-compacted powder sample which shows the isolated oxide and the interface oxide film particularly well.) Many of the larger

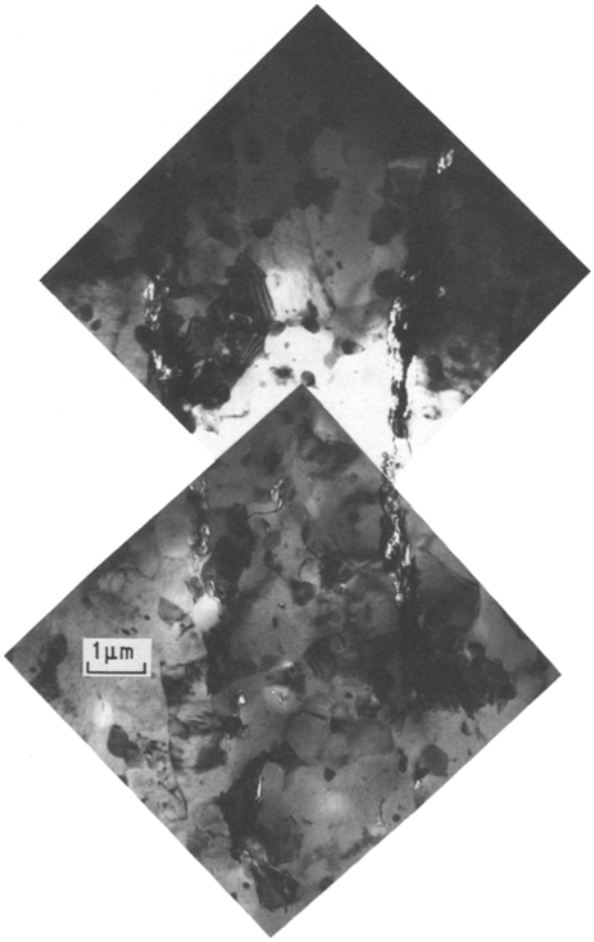


Figure 5 Thin zone of melting between two aluminium particles. The melted region consists of fine grains with dispersed oxide. Part of the interface between melted region and unmelted particle has a film of retained oxide.

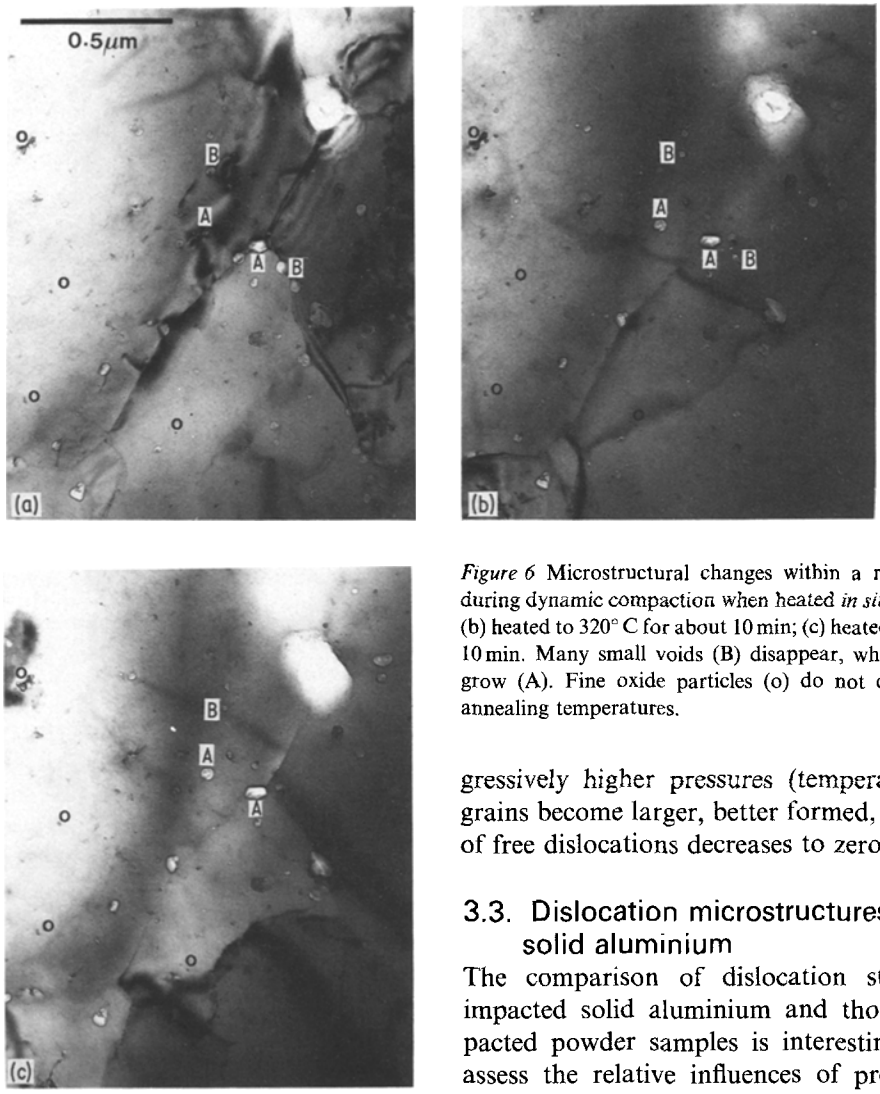


Figure 6 Microstructural changes within a region which melted during dynamic compaction when heated *in situ*: (a) as-compacted; (b) heated to 320° C for about 10 min; (c) heated to 450° C for about 10 min. Many small voids (B) disappear, whilst larger ones may grow (A). Fine oxide particles (o) do not change during these annealing temperatures.

melted regions contained a dispersion of fine oxide particles and voids (Fig. 6). In addition a number of high-angle boundaries and dislocations may be seen. Confirmation that these are sometimes thermally stable particles (oxides) and at other times voids (presumably shrinkage microporosity) is seen in the sequence of micrographs showing the changes taking place during *in situ* annealing. Many of the smaller voids (B) disappear on annealing, while some of the larger ones (A) grow. Such coarsening phenomena are particularly rapid for those voids which lie on boundaries, since these clearly act as rapid diffusion paths. It should also be noted how these voids can pin the boundaries, thereby inhibiting recovery or recrystallization processes. The many small oxide particles (o) remain unchanged during this annealing, even after higher temperatures in the microscope where eventual void and boundary disappearance occurred.

Dislocation rearrangement occurred after the initial high-speed deformation, which takes place as the shock wave passes. The extent of rearrangement into dislocation cells and subgrains varies with the pressure or temperature of the compaction shock. This is shown in Fig. 7 for materials prepared at a number of compaction pressures (temperatures). At low pressure (temperature) poorly formed, small subgrains are seen with a large number of free dislocations. At progressively higher pressures (temperatures) the subgrains become larger, better formed, and the number of free dislocations decreases to zero.

3.3. Dislocation microstructures in deformed, solid aluminium

The comparison of dislocation structures within impacted solid aluminium and those seen in compacted powder samples is interesting in helping to assess the relative influences of pressure pulse (or shear), adiabatic temperature rise, and duration of pressure or temperature. Fig. 8 shows typical microstructures in cold-worked (cold-rolled) aluminium samples, and in impacted solid aluminium. After cold rolling to 15 or 30% deformation, a dislocation cell structure is formed (Figs. 8a and b). Impact loading leads to a variety of structures according to the impact pressure and the duration of the high-pressure pulse. Low impact pressures lead to a high dislocation density, but little tendency for cell formation is seen (Fig. 8c). Much higher pressures, even though applied for very short times (1 μsec), or low pressures applied for relatively long times (5.9 μsec) give rise to reasonably well-formed cell structures (Figs. 8d and e).

4. Discussion

4.1. Microstructure and hardness of particle interiors

The particle interiors remaining after dynamic powder compaction are characterized here by a moderately high hardness, which can even show a slight increase on initially annealing before eventually, very slowly, recovering and softening. It is also seen that high compacting pressures lead to softer particle interiors.

The explanation of these results is found in the thermomechanical differences between powder compaction and solid-material working by rolling or by impacting, and corroborated by the different dislocation structures observed. The significant difference

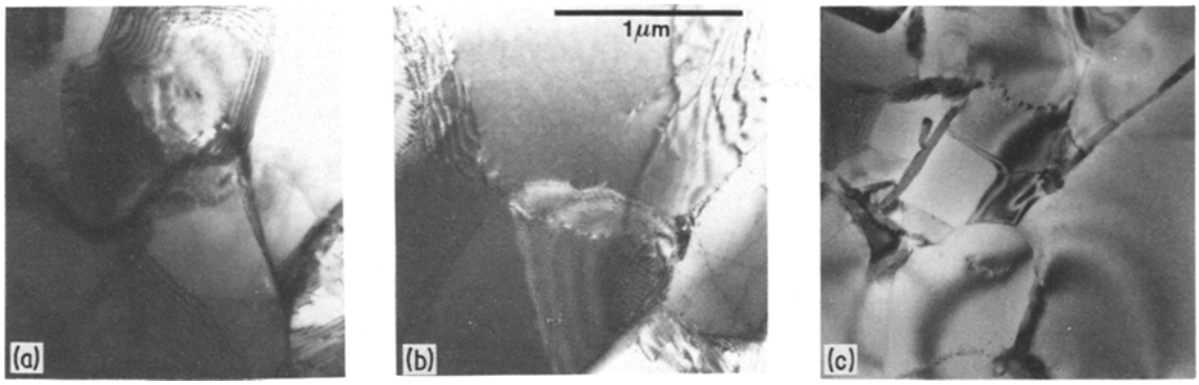
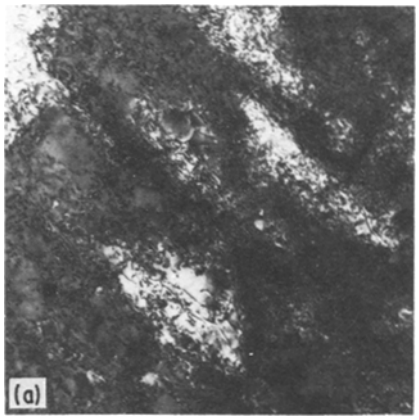
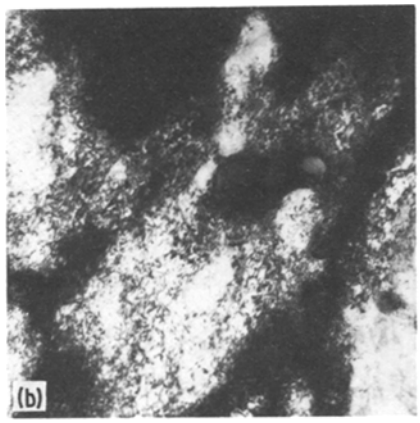
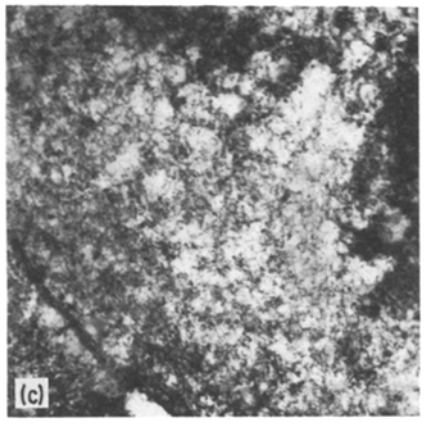


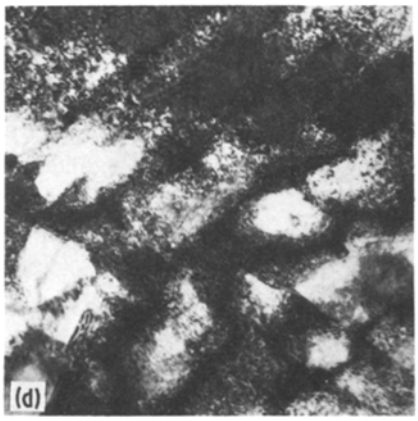
Figure 7 Dislocation structures within particle interiors after dynamic compaction. Compaction pressures (temperatures) were: (a) 1 GPa (130° C); (b) 3 GPa (400° C); (c) 4 GPa (530° C).

between compacted powder samples and impacted solid samples appears to originate principally in the time–temperature pulse seen. The adiabatic temperature rises and uniaxial strains which occur when solid material is impacted to the two pressures illustrated in Fig. 8 are about 30 and 120° C and 5 and 12% [7]. Furthermore, the residual temperature rise after pressure relief is much smaller (for example, a shock temperature rise of 120° C is followed by a residual temperature rise of only about 30° C [7]) and we can consider the high-temperature pulse duration to be the same as the high-pressure pulse duration, namely of the order of microseconds. During powder compaction, however, the temperature can easily rise significantly above 100° C (for the experiments reported here the temperature range about 100 to 530° C was covered), and this high temperature is maintained for long periods of time (seconds) since the deposited internal energy must be conducted completely out of the sample to the surroundings.









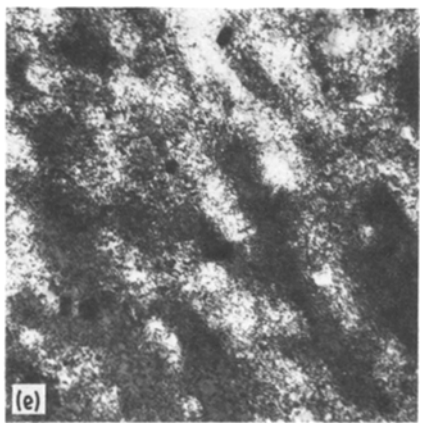


Figure 8 Dislocation structures in cold-worked and impact-loaded solid aluminium: (a) cold-worked, 15% strain and (b) cold- worked, 30% strain; (c) impact-loaded to pressure of 4 GPa for 1 μsec; (d) impact-loaded to 12.8 GPa for 1 μsec; (e) impact-loaded to 4 GPa for 5.9 μsec.

Cold-worked aluminium shows a poorly formed cell structure after high strain [8]. Pure aluminium more readily allows the formation of subgrains, but the presence of impurities and also inclusions, as found with commerically pure material, tends to leave the substructure in a less well-organized state [8]. These statements are fully in accord with the observation of poorly formed dislocation cells after cold working by rolling (Figs. 8a and b). A random arrangement of dislocations is expected after shock loading of aluminium at relatively low pressures [9], as seen in Fig. 8c, because the higher deformation rate, low adiabatic temperature and short time at higher temperature allow little time for dislocation rearrangement. Both higher-pressure impact-loadings (Fig. 8d) and longer shock durations (Fig. 8e) allow some dislocation arrangement to occur, and dislocation cell structures begin to appear. The material remains very hard after all these operations because of the high, relatively uniform dislocation density.

Following dynamic powder compaction the higher temperature and relatively long duration of this temperature pulse allow significant dislocation rearrangement, such that subgrains form. It is for this reason that the compacted powder samples are somewhat softer than the cold-worked material (Fig. 2) and become slightly softer at higher compression pressures (temperatures) (Fig. 3). The subgrains are larger and more perfectly formed after the higher-pressure consolidations (Fig. 7), in agreement with this conclusion. The reason for the slight increase in hardness of the compacted powder interiors on annealing at low temperatures (Figs. 2 and 3) is not clear. It is possible that the large number of point defects created during dynamic deformation still remain, in part, within the material and lead to more efficient hardening by point-defect aggregates after initial annealing. In addition, the higher thermal stability of the dynamically compacted material over that of the cold-worked material is not well understood.

Trueb [10], in a study of shock-loaded nickel, found differences in recovery and recrystallization behaviour between cold-worked and shock-loaded material, and also a variation with the impact pressures used. After shock loading at 7 and 32 GPa, he found a dislocation substructure similar to that of cold-worked material, but during annealing the substructure recovered without showing polygonization or recrystallization, in contrast to cold-worked material where both recovery and recrystallization were seen. A similar resistance to recrystallization during higher-temperature annealing would seem to explain the ability of the dynamically-compacted powder interiors to maintain relatively high hardness until high temperatures. Thus it seems that the recovery of the shock-loaded, random or cell dislocation structure (as exemplified in Figs. 8c to e) into uniform subgrain structures (as exemplified in Fig. 7) may induce a degree of stability against subsequent recrystallization.

4.2. Microstructure and hardness of melted zones

The melted zones observed in the dynamically compacted powder samples are characterized by an especially high hardness, a good resistance to softening even at high temperature, and a somewhat lower hardness for the higher compaction pressures.

The melted zones have been shown to resolidify very rapidly [3] and this leads to hardening by the very small grain size (see Figs. 4 and 5). Secondly, the oxide films from the original powder particles are broken into small pieces and dispersed, at least in part, throughout the melted zone. In addition, the shrinkage porosity caused by solidification is distributed as micropores, which contribute also to subgrain and grain-boundary retention and to hardening (Fig. 6). There are thus several contributions to the very high hardness of these melted zones, and the retained small grain size and oxide dispersion hardening can be taken as responsible for the high temperature stability.

It has been shown previously (e.g. [3]) that larger melted zones are produced when high pressure and temperature pulses are imposed during compaction. These larger zones resolidify more slowly, giving coarser structures, and there will be a more extensive post-compaction annealing. Softer materials are therefore to be expected following higher-pressure compaction.

5. Conclusions

Dynamic compaction of commercially-pure aluminium powder has been carried out using a number of compaction pressures, and the microstructures and hardnesses of the materials produced examined. In addition, cold-worked and impact-loaded solid samples have been examined in order to identify the relative effects of thermal and mechanical cycles on structure and properties. The thermal stability of the materials produced by dynamic compaction has been studied. The following major conclusions are made:

(i) Powder interiors are relatively hard after compaction and are able to retain significant hardness until very high temperatures. Higher compaction pressures lead to softer material.

(ii) Considerable recovery of the shock-induced dislocation substructure occurs during the later states of dynamic compaction, leading to well-formed subgrains. Higher compaction pressures correspond to higher adiabatic temperatures and more extensive recovery. Such recovery of the initially uniform dislocation structures appears to lead to a relatively recrystallization-stable structure.

(iii) The melted zones that are produced as pore-filling occurs during high-pressure dynamic compaction are extremely hard and highly resistant to softening at high temperatures.

(v) The melted zones possess a fine-grained structure following the rapid solidification of this material. In addition, these zones are strengthened by dispersed oxide from the initial powder surfaces, and by microvoids from the shrinkage occurring during solidification.

Acknowledgement

This work was carried out at Institut CERAC, Ecublens, Switzerland.

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Received 2 April
and accepted 21 May 1985