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Post-compaction heat-treatment response of dynamically-compacted Inconel 718 powder

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Dynamically-compacted Inconel 718 powder has been heat treated in order to examine the evolution of microstructural recovery and hardness, recrystallization and grain coarsening, and interparticle-adhesion and fracture response. Following dynamic compaction the compacted powders are imperfectly bonded and the material is fairly brittle; the shock wave has caused significant hardening; local microstructures and properties are variable from particle surface to interior. Following annealing at 900 to 1000° C, virtually complete recrystallization occurs giving rise to a material which is softened and has a fine-scale microstructure. Little improvement in fracture response occurs, however, because the oxidized prior-particle surfaces do not weld together. Shock-wave consolidation offers the possibility of producing monolithic, microstructurally-fine materials, providing suitable post-consolidation thermo-mechanical processes are developed to overcome the limited bonding problem.

1. Introduction
The shock wave processing of materials is interesting in order to (i) consolidate powders [1–3] and (ii) pretreat material for subsequent improved behaviour, for example activated sintering [4] or enhanced mechanical or precipitation response [5]. The present report describes the use of dynamic powder compaction to consolidate Inconel 718 powder, and the evolution of structure and properties during post-compaction heat treatment.

Dynamic powder compaction has been shown to have potential for the consolidation of metastable and very-fine-scale materials. The process often gives rise to poor or irregular bonding between powder particles and to inhomogeneous microstructures [6]. In addition a certain porosity may remain. Simultaneously, the passage of the intense shock wave causes significant hardening [7], with possible associated advantages.

This study examines the extent of thermal treatments necessary in order to obtain homogenization of the material microstructures. At the same time, the extent of inter-particle bonding is assessed by fracture studies. In particular, the question of whether such homogenization and efficient bonding can be achieved at low enough temperatures to avoid significant structural coarsening or irrecoverable property losses is addressed.

2. Experimental techniques
The powders used were spherical, gas-atomized powders with a mean size of about 100 μm. Dynamic compaction tests were performed using a two-stage gas gun to launch nylon pistons onto the powders, held within a high-strength pressure container at the end of the launch tube. The details of compaction procedure, and methods for deducing the shock pressures using the impedance-matching approach, have been described previously [2, 6]. The particular materials studied here were prepared using impact velocities of 1500 or 2000 m sec^{-1} which gave rise to compaction pressures of 5 or 8 GPa, respectively.

Optical metallography was performed on longitudinal sections through as-compacted samples, or after heat treatments at 800 to 1000° C for 1 h, followed by air-cooling. These treatments were intended to homogenize and sinter the compacted materials and no particular attention was given to mechanical property optimization by subsequent precipitation. Microhardness testing was carried out on polished sections using a Leitz Vickers hardness tester with loads of 200 and 25 g. Transmission electron microscopy was performed using a Philips 300 microscope on thin foils prepared using a perchoric acid–ethanol electropolishing solution. Finally, small, transverse samples were fractured by bending such that crack propagation was in the same direction as the shock wave travel: fracture surfaces were examined using a Cambridge Stereoscan 250 microscope.

3. Results and discussions
3.1. General microstructure and microhardness
Typical microstructures of as-compacted and heat treated samples are shown in Fig. 1. In the as-compacted samples the original dendrite structure of the powder particles is clearly distinguished, in addition to the regions of inter-particle melting (m) which form during compaction. The fraction of melting occurring is estimated, from such micrographs, to be of the order of 10 and 20% for compacting pressures of 5 and 8 GPa, respectively.

After annealing at 800° C, no change is evident in the optical micrographs. After annealing at 900° C the melted zones are more difficult to distinguish and have taken a fine, microcrystalline appearance: in addition the material contains a distribution of fine precipi-

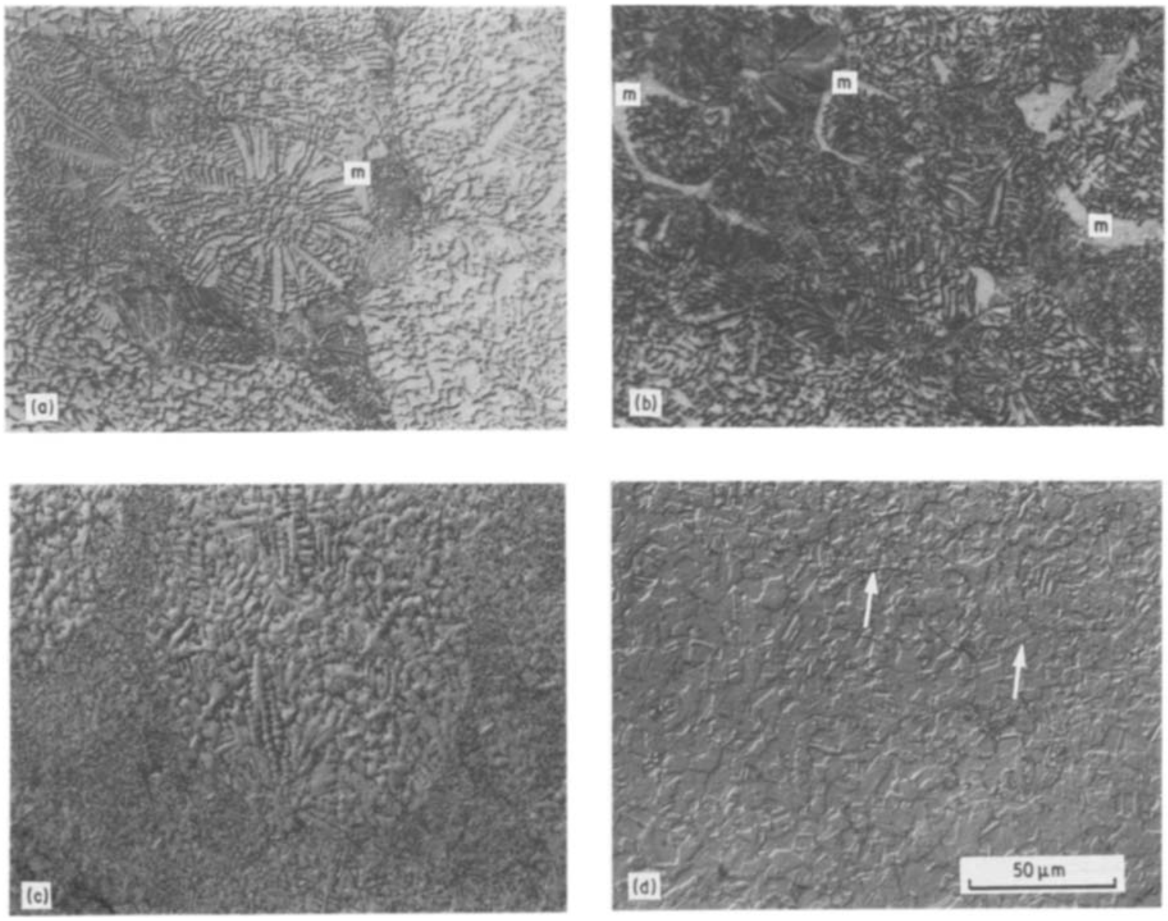

Figure 1 Optical micrographs of dynamically-compacted Inconel 718: (a) as-compacted, 5 GPa; (b) as-compacted, 8 GPa; (c) compacted at 5 GPa and heat-treated at 900° C for 1 h; (d) compacted at 5 GPa and heat-treated at 1000° C for 1 h. Regions which melted during compaction are indicated (m). Prior particle boundaries remain sometimes visible (arrows) after annealing at 1000° C.

tates. After annealing at 1000° C the material appears totally recrystallized and essentially homogeneous. At certain locations the prior-particle boundaries remain visible, however, indicating their decoration by surface contamination. Many of the fine particles observed after annealing at 900° C have dissolved at 1000° C.

The microhardness results obtained on as-compacted material, and also after annealing treatments, are shown in Fig. 2. Samples compacted at 5 and 8 GPa gave essentially the same results and no distinction is made here. The smaller load allowed the separation of measurements on melted zones and on particle interiors, which showed the melted zones to be distinctly harder and to retain this hardness differential even after heat treatments at 800 and 900° C. No evidence of these melted zones remained after annealing at 1000° C.

The large amount of shock hardening occurring during dynamic compaction is evident. The additional hardness of the melted zones is similar to that observed on dynamically compacted aluminium powder [6], but was not observed for the explosive compaction of Mar-M200 superalloy powder [7]. Similar softening after annealing aged and shock-hardened material in the range 800 to 1000°C was observed by Meyers and Orava [5] and associated with the coarsening and dissolution of γ'' precipitates and with recrystallization (between about 900 and 950° C).

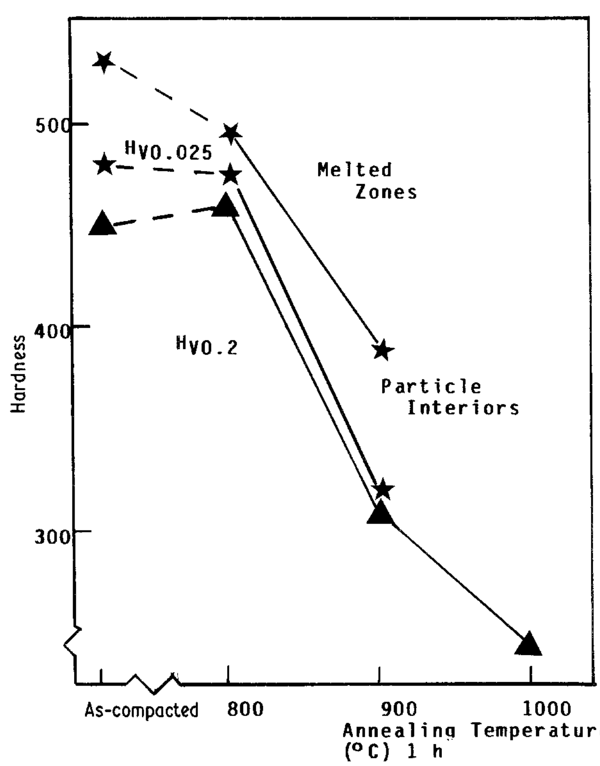

Figure 2 Microhardness results on as-compacted and annealed Inconel 718. The data obtained using the 25 g load are separated into those measurements on melted zones and those on particle interiors: no melted zones were distinguished after heating at 1000° C. ▲ = 200 g load; ★ = 25 g load.

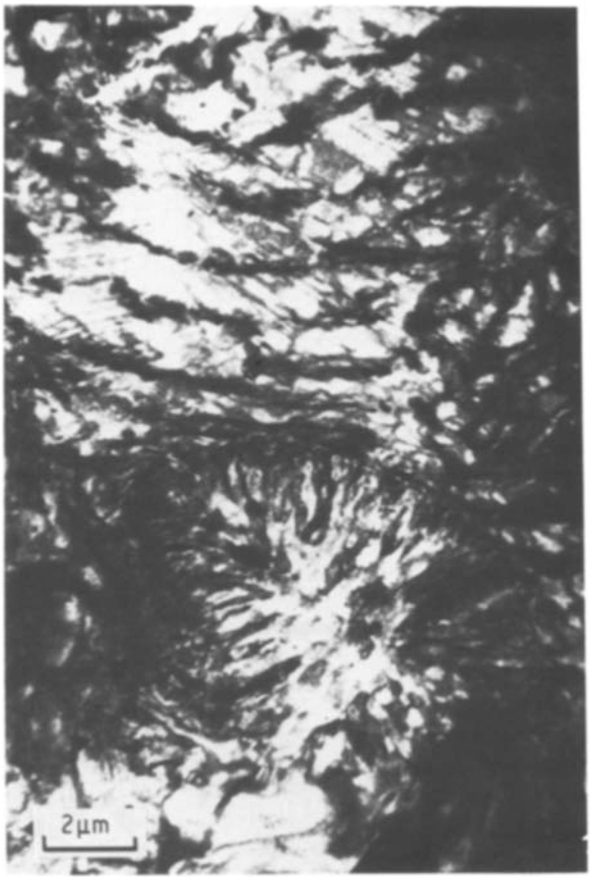

Figure 3 Transmission electron micrograph illustrating the typical deformation structure, with associated dendrite wall contrast, and melted zone, within as-compacted Inconel 718.

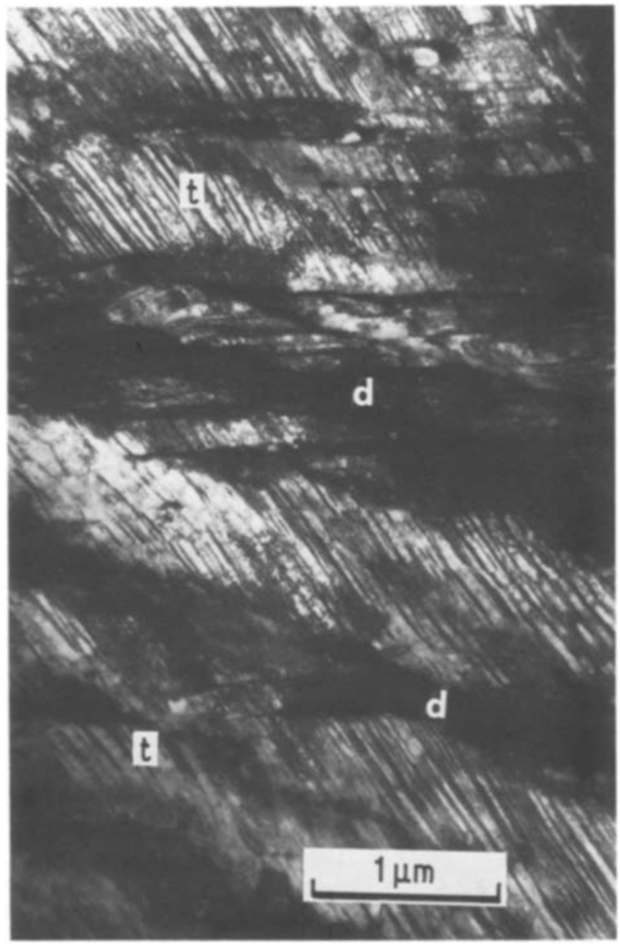

Figure 4 Transmission electron micrograph showing the high twin density associated with the shock deformation. Twins are often bent (t) because of the high strain incurred. The locally-high dislocation density at the original dendrite walls (d) is visible.

3.2. Transmission electron microscope results

Fig. 3 shows a melted zone at the interface between two powder particles in as-compacted material. (Note that, apart from the fraction of melted material, the samples compacted at 5 GPa and 8 GPa showed no differences, and no further distinction will be made here). Resolidification of the molten material has taken place from the solid substrate giving rise to columnar grains which meet along the centre-line of the melted volume. The particle interiors are heavily deformed, showing twinning, with additional contrast due to the dendrite walls of the powder particles. Fig. 4 shows these dendrite walls in more detail, the high density of shock-induced twins, and the local concentration of dislocations at the dendrite walls. The twins are fairly uniformly distributed, with a spacing of about 50 to 100 nm, and often appear bent because of the high strain which the material has suffered.

Fig. 5 shows that recrystallization has already started at 800° C, but is limited principally to the interparticle boundaries where no significant melting has occurred. Only minor relaxation of dislocation or deformation-twin structure occurs in agreement with the observed maintenance of high hardness. The interparticle boundary is clearly indicated, after recrystallization, by the line of carbides. At other locations along particle boundaries, Fig. 6, recrystallization is hardly beginning, and small recrystallized grains and carbide particles are seen. The melted zones do not yet recrystallize, Figs 5 and 7, despite the small grain size and high dislocation density, except for some regions close to a recrystallizing area (arrow in Fig. 7): in this case the recrystallization front begins to penetrate the melted zone. The recrystallized grain size remains small and the grain boundaries are frequently coated with coarse carbide particles: these particles may originate in the solute segregation of the dendrite walls.

Microstructures observed after annealing at 900° C for 1 h are shown in Figs 8 to 10. Fig. 8 shows that most of the material has in fact recrystallized (based on the TEM observations about three-quarters of the material is estimated to have recrystallized). Carbide particles from the original dendrite walls and from the powder particle surfaces restrict the grain size to a few micrometres. Such carbides, as well as the deformation twins, are effective at restricting the propagation of the recrystallization front into the non-recrystallized areas, Fig. 9. The melted zones still remain essentially non-recrystallized, Fig. 10.

Occasionally recrystallized grains are seen here, and the entire zone appears well-relaxed with a fairly equiaxed grain morphology and a lowered dislocations density. The melted zones often appear to contain very fine precipitates, as illustrated in Fig. 10. It is not clear whether these are fine precipitates which form following the dissolution-quench cycle involved during melting, or are dispersed surface contaminants from

Figure 5 Microstructure of dynamically-compacted Inconel 718 after heat treatment at 800° C for 1 h. Powder particle boundaries show the beginnings of recrystallization (a), and the boundaries remain decorated with coarse carbide particles. The melted zones (b) formed during compaction do not recrystallize. Particle interiors still show the high dislocation and twin densities (c).

powder particle surfaces (similar to the oxides dispersed within the melted pools of compacted aluminium powder [6]).

Microstructures seen after annealing at 1000° C are shown in Figs 11 and 12. The material is now virtually completely recrystallized, Fig. 11, with a grain size of about 4 μm. Many of the carbides have dissolved, and even though the linear arrangement of carbides can still be distinguished, these are ineffectual at preventing grain coarsening. Occasionally melted zones remain non-recrystallized, Fig. 12. In these cases an intense distribution of stable (presumably oxide) particles is seen which is clearly responsible for maintaining the dislocation and grain structure.

3.3. Fracture surface studies

The fracture surfaces of samples broken in the as-compacted state, and also after heat treatments at 900 and 1000° C are shown in Fig. 13. The micrographs shown all correspond to the material compacted at 5 GPa. The material compacted using 8 GPa was similar but contained more large-scale cracks as a consequence of the over-violent pressure relief. Material behaviour after annealing at 800° C was essentially the same as that seen on the as-compacted material.

About one half of the fracture surface of the as-compacted material (Figs 13a and b) shows fine dimples characteristic of ductile fracture. These regions correspond to the areas where effective interparticle bonding has been achieved, either by melt formation or by solid-state-friction type welding. In addition some long interparticle cracks were seen, indicative of very poor local bonding. On some occasions the powder particles have adopted the characteristic upper-convex, lower-concave morphology observed by optical microscopy [2]. The smoother particle surfaces correspond to the areas where highly-imperfect bonding has been achieved. In some of these areas the dendritic-surface appearance of the original powders is seen (Fig. 13b), after being deformed by the intense surface shear occurring during compaction.

Following annealing at 900 and 1000° C the changes

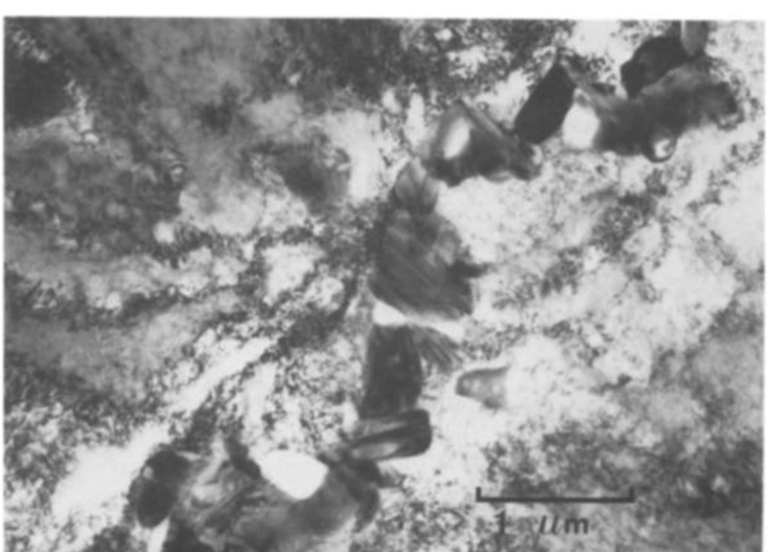

Figure 6 Microstructure of dynamically-compacted Inconel 718 after heat treatment at 800° C for 1 h. Recrystallized grains and carbides are beginning to form along the powder particle interfaces.

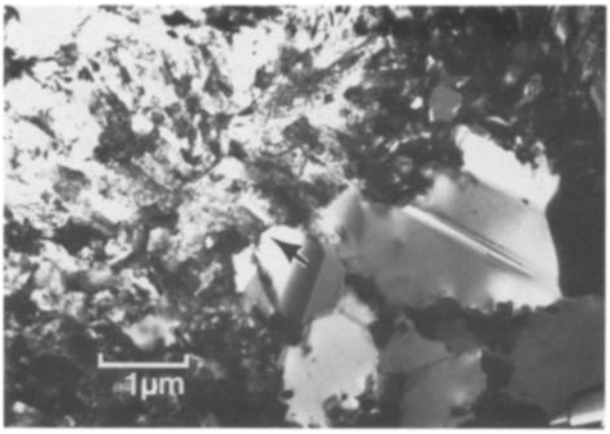

Figure 7 Non-recrystallized melted zone, with nearby recrystallization outside the melted zone: Inconel 718 after dynamic compaction and heat treatment 1 h, 800° C. The recrystallized grains start to grow in to the melted zone (arrow). Carbides restrict the growth of the recrystallized grains.

in fracture morphology are rather minor. About the same, one half of the fracture surface has a ductile appearance; the same, occasional inter-particle cracks are seen. The general surface morphology becomes somewhat less angular as the powder particle facets slightly lose their planar nature.

Other, subtle changes are evident in the higher-magnification micrographs (Figs 13b, d and f): the ductile fracture areas show a finer scale of dimples; the smooth, unbonded areas remain smooth apart from the appearance of fine particles at 1000° C (Fig. 13f). In addition, after annealing at 1000° C, a small number of clusters of large particles was found on the fracture surface.

These observations reflect the changes in microstructures, particularly those occurring at particle boundaries – softening by recrystallization and by carbide coarsening and subsequent dissolution – which allows a more ductile failure of the bonded regions. The imperfectly-bonded particle surfaces do not show any significant improvement in bonding by the high temperature anneals. These poorly-bonded areas have a large number of fine particles which may be associated with the impurities found on the original particle surfaces which play an important role on inhibiting particle bonding.

4. Summary and conclusions

Dynamic compaction of the powders studied here has led to the creation of a high-density, partially-bonded material. The remnant porosity, and particularly the regions of poorly-bonded interparticle interfaces,

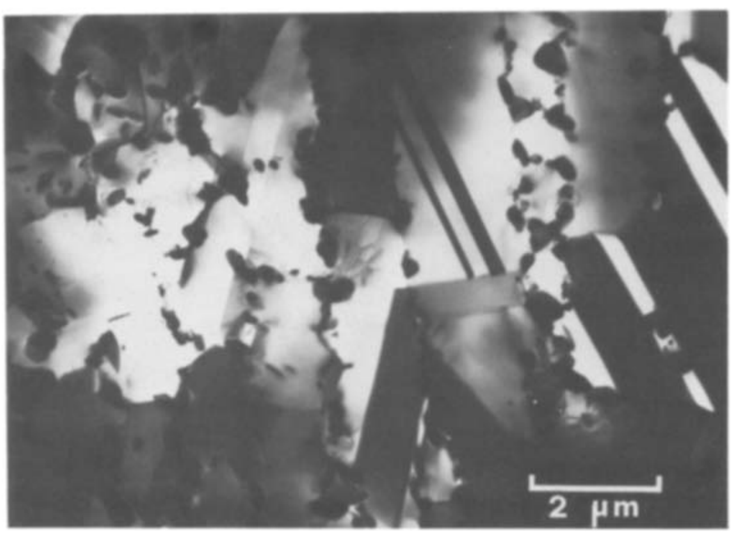

Figure 8 Well-recrystallized region within the compacted Inconel 718 after heat treatment at 900° C for 1 h. Carbides from the original dendrite walls restrict the growth of the grains.

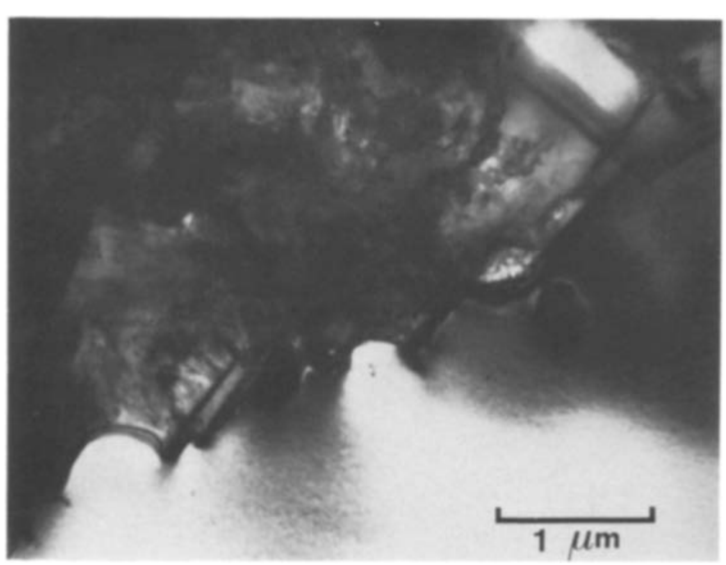

Figure 9 Motion of the recrystallization front is restricted by carbides and deformation-twin platelets: Inconel 718 after heating at 900° C for 1 h.

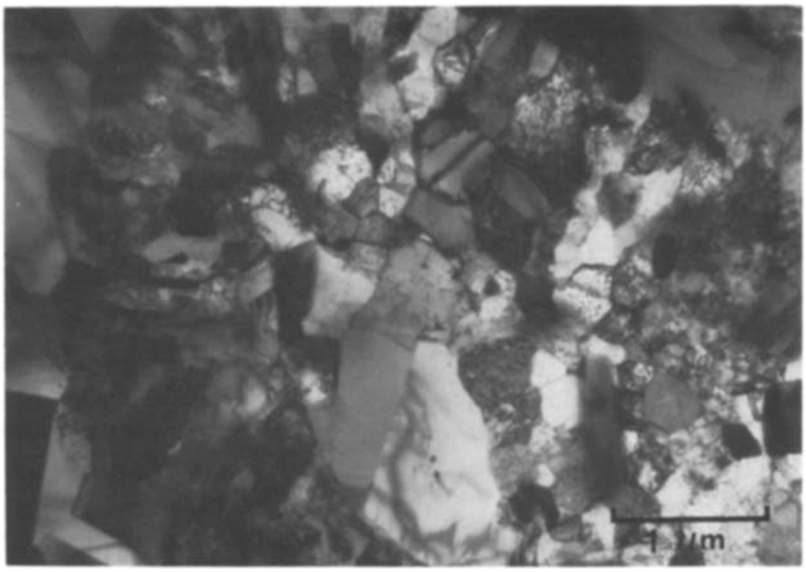

Figure 10 Melted zone within dynamically-compacted Inconel 718 after heating at 900° C for 1 h. Little recrystallization has occurred and the structure appears well-relaxed. A fine precipitation is evident within the non-recrystallized area.

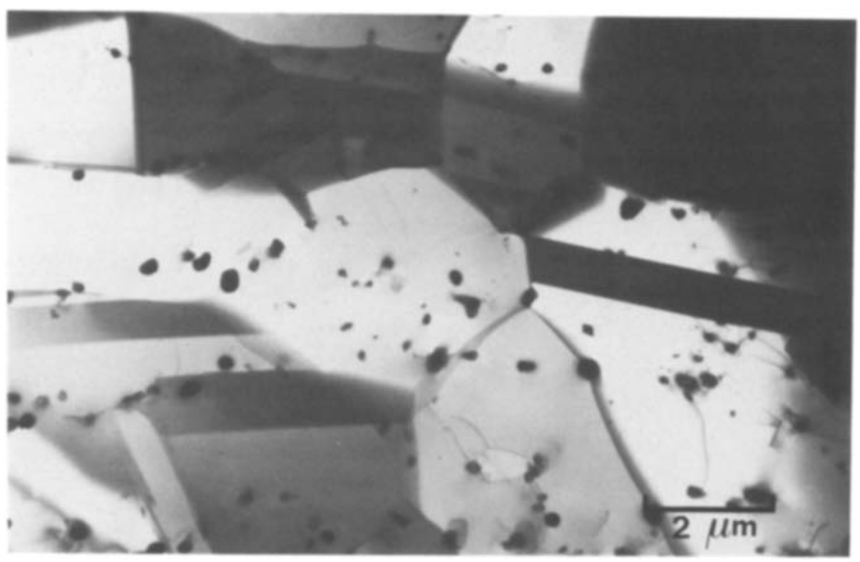

Figure 11 Well-recrystallized region typical of material heat treated at 1000° C. Carbide particles are slowly dissolving at this temperature, but the remnants of the linear distribution of particles is still visible.

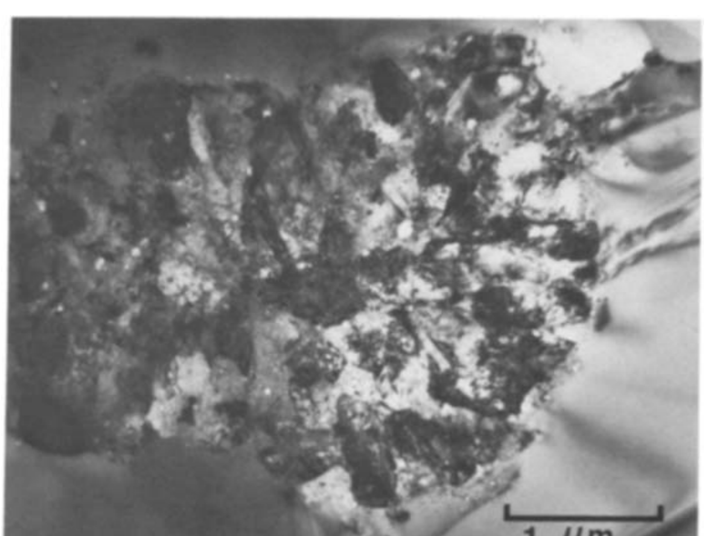

Figure 12 Non-recrystallized melted zone after heat treatment at 1000° C. The region is stabilized by fine particles, presumably oxide from original powder particle surfaces.

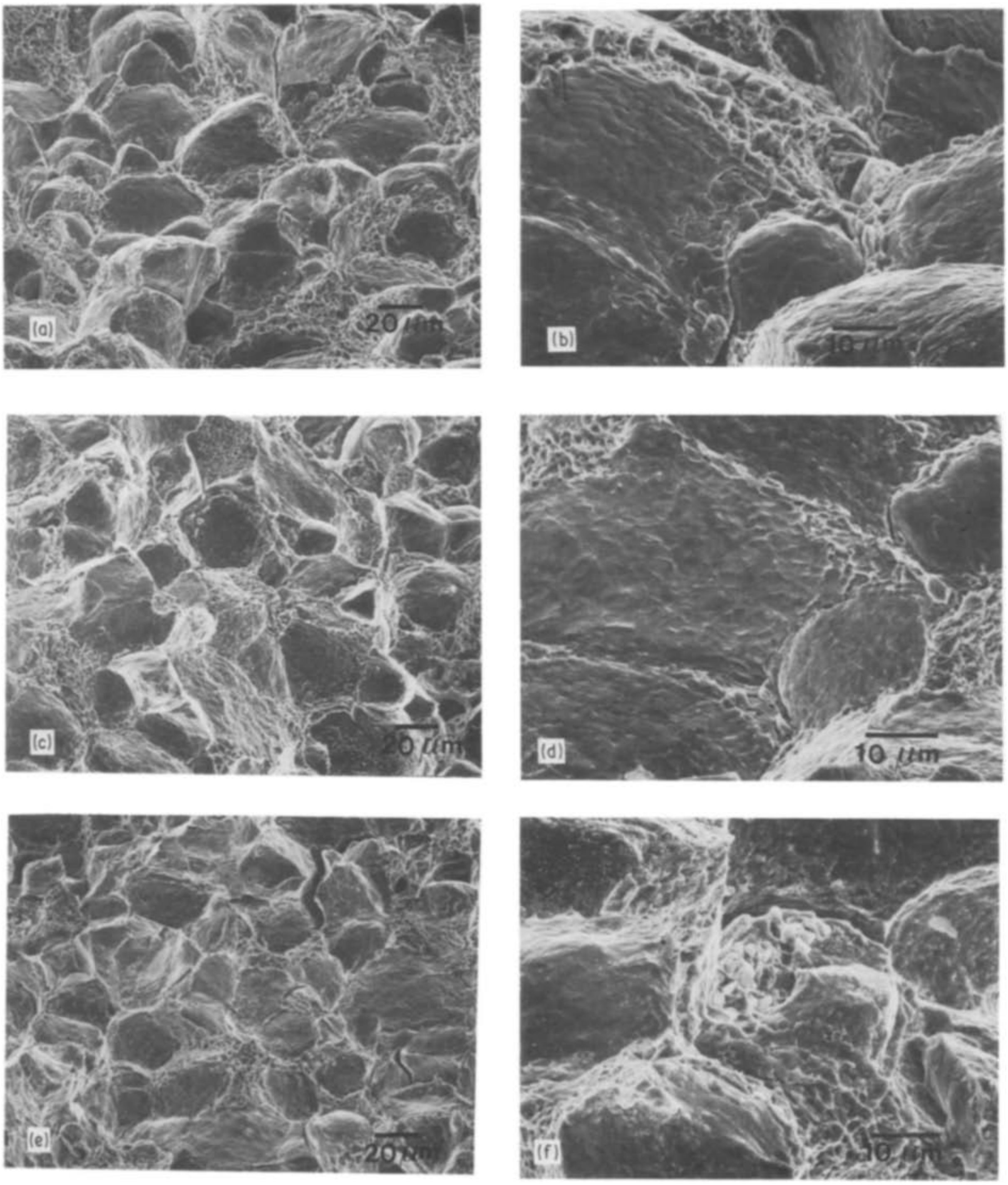

Figure 13 Fracture surfaces of: (a, b) as-compacted Inconel 718; (c, d) after heat treatment at 900° C; (e, f) after heat treatment at 1000° C. Compaction pressure used was 5 GPa. In the as-compacted state some of the powder particles clearly have an upper convex surface/lower concave surface.

make subsequent processing essential for this material. During shock wave passage, a very high dislocation and twin density is created. In addition to the hardening produced by these defects, the high defect density would be beneficial for obtaining a fine and uniformly-distributed subsequent precipitation: it has previously been shown that the γ'' precipitate forming in this alloy has a high misfit with the lattice and much more uniform precipitate nucleation can be achieved following intense working or shock deformation [5]. Such defect benefits can only be utilized provided that no post-compaction processing requires such high temperatures that the dislocation and twin structures are lost. Following compaction the microstructure, and correspondingly the local mechanical property (microhardness), is inhomogeneous: while the particle interiors are fairly uniformly shock-hardened, there is generally a concentration of deformation near the powder particle surfaces; at certain locations the adiabatic deformation has been sufficiently intense that local heating occurs leading to relaxation, recrystallization, and even to melting. Complete homogenization of the microstructures is only obtained after full recrystallization, requiring heat treatments in the range 900 to 1000° C (ignoring for the moment those melted zones which are oxide-particle stabilized and still not recrystallized after heating at 1000° C). The partially-recrystallized structures correspond to unsatisfactory materials because of the local softening at particle surfaces where the recrystallization preferentially starts.

The imperfect bonding achieved here by dynamic compaction has not been improved in any obvious way even after heating for full recrystallization at 1000° C. The reason for the inefficient bonding, and the lack of visible improvement on heat treating, appears to be associated with the contaminant layer on the original powder particle surfaces (from powder preparation or subsequent handling): the evidence for this is the large number of fine particles observed on the unbonded particle surfaces after fracture and the melted pools formed during compaction which contain the dispersed debris from the original powder particle surfaces. For this material, a fully-bonded monolith would only be prepared by combining the present dynamic compaction conditions with more effective thermo-mechanical processing or by using more violent dynamic compaction conditions. The present tests have not shown significant improvements in material microstructure or properties when using 8 GPa compaction pressure instead of 5 GPa: while significantly more melting is achieved, difficulty in controlling the relief of the shock pressure means a greater tendency for cracking after the higher pressure compaction. Further development should consider the combination of "medium-pressure" dynamic compaction, useful for achieving a high density and medium-level bonding, with subsequent medium-temperature thermo-mechanical processing: the high defect density caused by shock-wave processing could enhance sintering or precipitation kinetics; mechanical working, for example forging, is important during the second stage of processing to ensure break-up of contaminant films and thereby to achieve complete bonding.

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