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**Rapidly Solidified Materials: Properties and Processing**  
**Proceedings of the 2nd International Conference on**  
**Rapidly Solidified Materials**  
**San Diego, California, 7-9 March 1988**

Published by 

**8802-005**

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## SHOCK CONSOLIDATION OF RAPIDLY SOLIDIFIED TITANIUM ALLOY POWDERS

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### ABSTRACT

Consolidation of rapidly solidified titanium alloy powders employing explosive shock pressure was carried out successfully. The cylindrical axi-symmetric explosive consolidation technique was utilized, and compacts with a density above 95% were produced. Better consolidation (with more interparticle melting regions and less cracking) was achieved by using a double tube design in which the outer tube (flyer tube) was explosively accelerated, impacting the powder container. Optical and transmission electron microscopy observations were carried out to establish microstructural properties of the products. It was observed that consolidation is achieved by interparticle melting occurring during the process. The interior of the particles in Ti-17 alloy exhibited planar arrays of dislocations and twin-like features characteristic of shock loading. Two dominant types of microstructures (lath and equiaxed), were observed both in Ti-662 and Ti-6242 + 1%Er compacts, and very fine erbia ( $\text{Er}_2\text{O}_3$ ) particles were seen in the latter alloy. The micro-indentation hardness of the consolidated products was found to be slightly higher than that of the as-received powder material; and the yield and ultimate tensile strengths were found to be approximately the same as in the as-cast and forged conditions. These superior mechanical properties are a consequence of strong interparticle bonding between individual powder particles. It was also established that scaling up the powder compacts in size is possible and compacts with 50, 75, and 100 mm diameter were successfully made.

SHOCK CONSOLIDATION OF RAPIDLY solidified metals is a process that has considerable potential. Consolidation can be achieved without prolonged exposure of the compact to high temperatures.

The shock wave traveling through the powder promotes melting of the powder surface layers. This molten material is an effective bonding agent and is rapidly resolidified after the passage of the shock pulse. Considerable work has been carried out on shock consolidation of rapidly solidified materials [1-7], but many roadblocks exist before this technology can be industrially implemented. The main problem is cracking of the compacts at both the microscopic and macroscopic levels. The three principal objectives of this investigation were (a) to demonstrate that rapidly-solidified titanium alloy powders can be consolidated by shock waves, (b) to establish whether the process can be scaled up, and (3) to determine the mechanical properties and microstructural characteristics of the shock-consolidated alloys.

### MATERIALS AND TECHNIQUES

Three titanium alloys (REP Ti-17, PREP Ti-662, and RSR Ti-6242 + 1% Er) were used in this study. They were produced by three different but closely related techniques; rotating electrode process (REP), plasma rotating electrode process (PREP), and rapid solidification rate process (RSR). Ti-17 and Ti-662 powders were obtained from Nuclear Metals Corp., while Ti-6242 + 1% Er was obtained from Pratt and Whitney Aircraft Group of United Technologies. These titanium-based alloys were supplied by General Electric. Table 1 shows the nominal compositions of the three titanium alloys. The powder sizes ranged from 250 to 50  $\mu\text{m}$  and they were spheroidal in shape. The Ti-6242 powder, prepared by the RSR technique, had a greater amount of satellites than the other two powders. The structure of the powders was microdendritic or microcellular. Table 1 shows the nominal compositions of the three titanium alloy powders. The apparent densities of the powders were approximately 60-70% of the theoretical density.

Table 1. Nominal Compositions of Three Titanium Alloy Powders  
Chemical Composition %

Material	Al	Sn	Zr	Mo	Cr	V	Ti	Cu	Er	Ti
Ti-17	5.0	2.0	2.0	4.0	4.0	--	--	--	--	bal.
Ti-662	6.0	2.0	--	--	--	6.0	1.0	1.0	--	bal.
Ti-6242	6.0	2.0	4.0	2.0	--	--	--	--	1.0	bal.

For shock consolidation, the cylindrical geometry was used in this study. Two configurations were used: (a) single tube in which the explosive is in direct contact with the powder container (Figure 1(a)); (b) double tube in which the powder container is shielded with another tube acting as a flyer tube (Figure 1(b)). The single tube technique is amply described in the literature [e.g., ref. 8], while the flyer-tube technique has recently been introduced [9]. The explosive is detonated at the top and causes the implosion of the tube containing the powder, as the detonation front runs downward. In the flyer-tube technique, the flyer tube impacts the powder tube, and the pressures generated are considerably higher than in the single-tube technique. A central mandrel was used along the cylinder axis to trap the Mach stem. Pressure calculations are given by Coker [10]. The explosives used were ANFO and TNT and the detonation velocities ranged from 3,000 to 3,700 m/s for ANFO and 3,500 to 4,500 m/s for TNT. Standard metallographic techniques were used for the preparation of specimens for optical and electron microscopy. Tensile tests were conducted at General Electric Aircraft Engine Division at ambient and high temperatures. The specimens were cylindrical with threaded ends. Their diameter (in the reduced section) was 4 mm, the gage length was 20 mm and the initial overall length was 50 mm. They were machined so that the specimen axis was parallel to the compacted cylinder axis. The ends were shot-peened in order to decrease the tendency for fracture initiation in the threads.

## RESULTS AND DISCUSSION

**MICROSTRUCTURE** - Twenty one consolidation experiments were conducted in this investigation. Generally, the powder particles underwent a considerable amount of deformation. This is illustrated in Figure 2, that shows a particle that was subjected to shock consolidation without bonding (insufficient pressure for jetting and melting between particles).

Single tube compaction yielded compacts with considerable density of voids and cracks. The differences are clearly evident in Figure 3. One of the regions that underwent melting is indicated with an arrow. This improved bonding

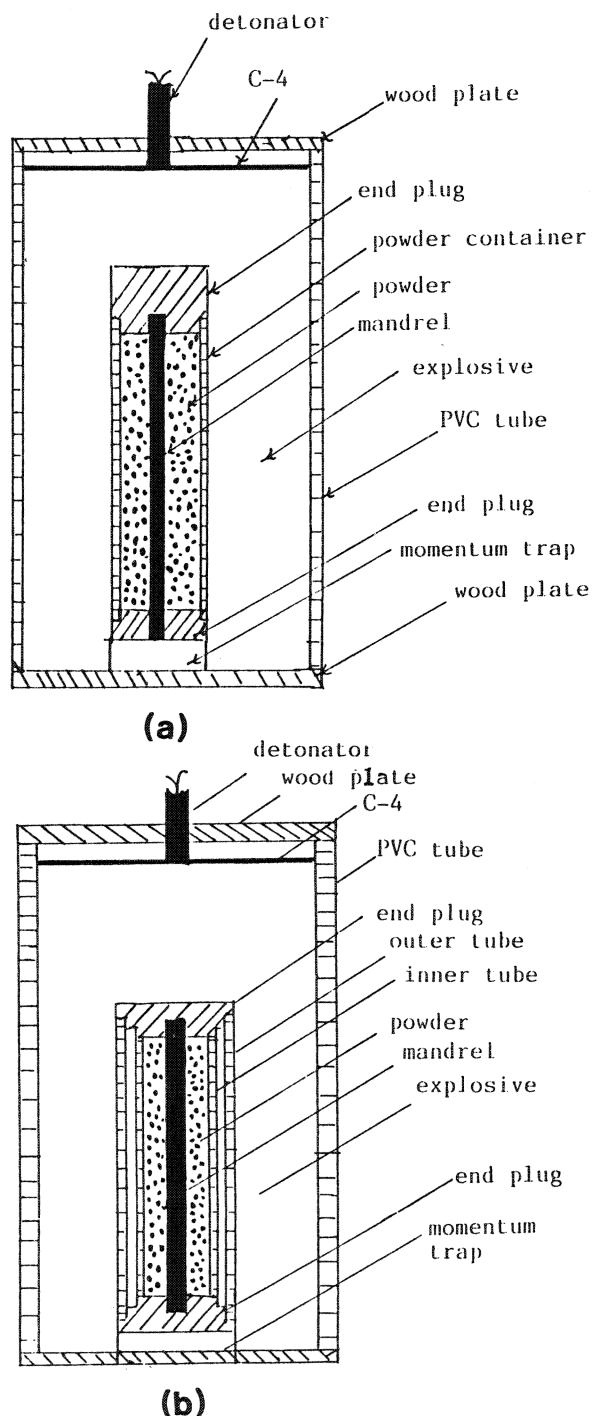


Fig. 1 - Systems used for shock consolidation of titanium alloys; (a) single tube; (b) double (or flyer) tube.

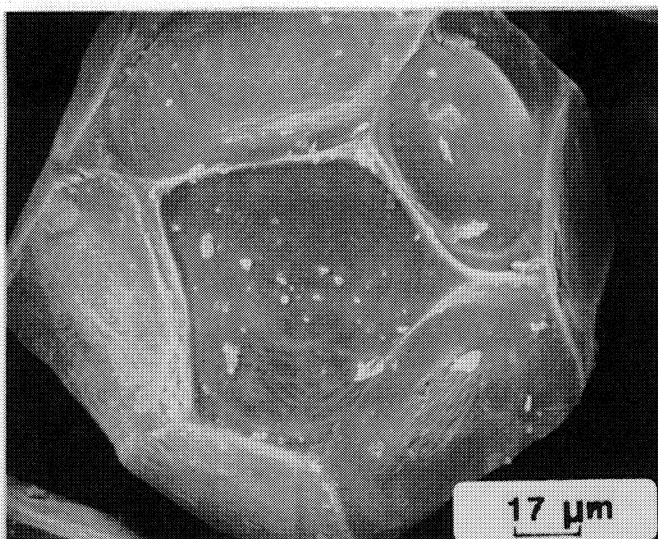
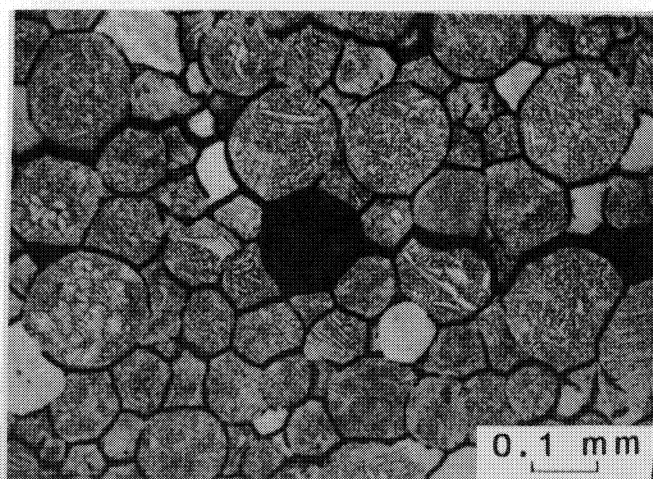
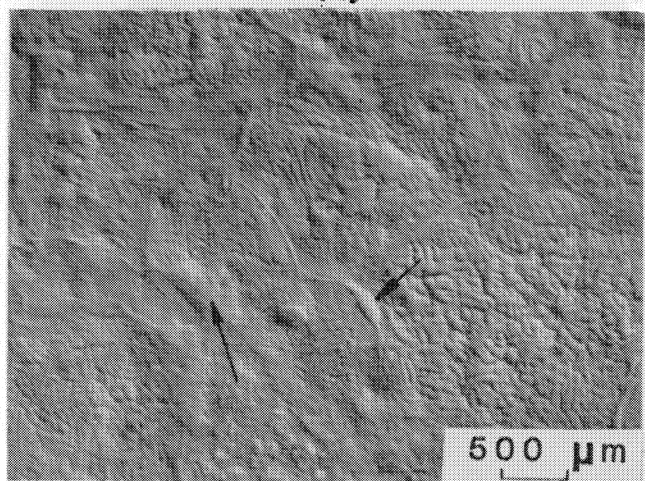


Fig. 2 - Scanning electron micrograph showing that powder particles underwent a considerable amount of deformation; the particles were initially spherical.



(a)



(b)

Fig. 3 - Comparison of structures between compacts of Ti - 17 obtained with (a) single-tube and (b) double-tube technique.

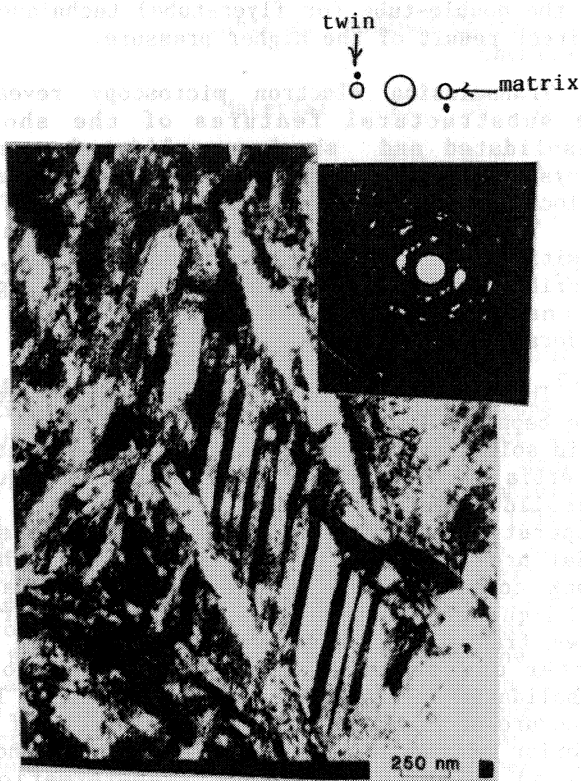
for the double-tube (or flyer-tube) technique is a direct result of the higher pressure.

Transmission electron microscopy revealed the substructural features of the shock-consolidated and shock-consolidated + aged alloys. Figure 4 shows a triple point and dense dislocation arrays in the three grains of Ti - 17. These arrays of dislocations in high densities are typical of shocked materials, as described by Meyers and Murr [11]. This region is characteristic of the interiors of the powders.

The titanium alloys strengthened by erbia have been the object of considerable study. The rapid solidification process allows the retention of erbia as very small dispersions; subsequent consolidation at ambient or moderately low temperatures will retain this fine dispersion (that provides good creep resistance). Thus, shock consolidation has been envisaged as a technique to accomplish this goal. Figure 5 shows transmission electron micrographs of the center and particle interfaces for shock-consolidated Ti-6242 + Er alloy. The dense lath structure of Figure 5(a) is characteristic of the interior of the particles and is due to shock-induced twinning and phase transformations.



Fig. 4 - Transmission electron micrograph showing planar arrays of dislocations in Ti-17.



(a)



(b)

Fig. 5 - Transmission electron micrograph showing (a) a lath structure with high dislocation and twin densities and (b) fine equiaxed grains and  $\text{Er}_2\text{O}_3$  dispersoids.

Twinning is evident from twin spots in the diffraction pattern. The substructure of Figure 5(b) is very different. It shows small equiaxed grains with an approximate size of  $0.05\ \mu\text{m}$ . Because of the very low dislocation density, it is believed that this region underwent either melting or recrystallization. Some  $\text{Er}_2\text{O}_3$  particles can be seen; they are marked with arrows. In order to reveal the erbia particles more clearly, and to demonstrate that the consolidation process did not significantly increase their size, annealing treatments of shock consolidated alloys were conducted, followed by TEM observations. The substructure after two hours at annealing at  $870^\circ\text{C}$  is shown in Figure 6. The  $\text{Er}_2\text{O}_3$  dispersoids are clearly seen. Their size and spacing is such that they are effective high temperature strengtheners. There seems to be a tendency for them to arrange themselves in rows.

The observation of Ti-662 revealed the same features as the other two alloys: (a) regions containing laths/twins and high dislocation densities and (b) regions with equiaxed grains.

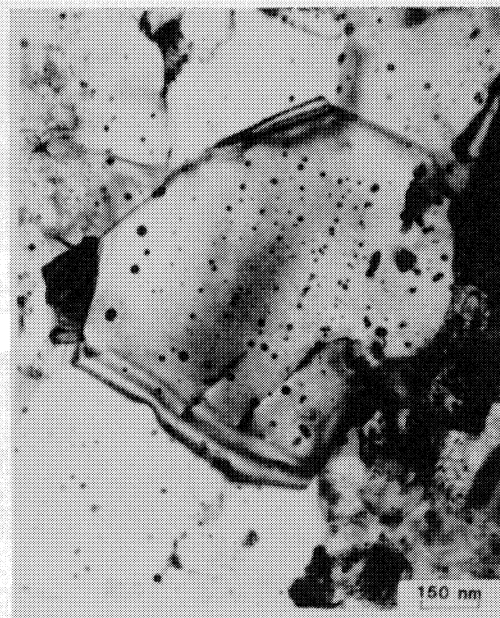


Fig. 6 - Transmission electron micrograph of shock consolidated Ti-6242 + Er after two hours annealing at  $870^\circ\text{C}$ .

**MECHANICAL PROPERTIES** - The hardness of the as received Ti-17 powder increased considerably from 325 HV100 in the as received condition to 500 HV200 in the consolidated condition. Similar results were observed on Ti-662 powder (362 HV200 to 480 HV200) and Ti-6242 + 1% Er powder (from 413 HV100 to 435 HV200). Micro-indentations were made on cross sections cut toward the top and bottom parts of each compact. The hardnesses were found to be uniform throughout the cross-section. The microhardness of Ti-662 compacts after aging heat treatment was also measured and was found to be lower than that of as compacted condition.

Tensile tests carried out on consolidated Ti-17 after aging heat treatment conditions showed better strength properties when compared with the as-cast material. The average 0.2 percent yield strength and ultimate tensile strength were 1215 MPa and 1233 MPa, respectively, for the shocked and aged alloy. In contrast, the average yield strength and ultimate strength of the as-cast Ti-17 alloys are generally around 1069 MPa and 1138 MPa, respectively [12]. The ductility of compact was poor compared to the as-cast Ti-17 alloys.

The tensile strengths of the Ti-662 compacts were slightly lower than the standard forged specimen (Figure 7(a)). The ductility of the compact in the as received condition was very poor compared to the standard forged specimen (Figure 7(b)). However, after 870°C and 2 hours HIP process, the ductility at room temperature was better than the standard forged specimen.

The lower strength, compared to the standard forged specimen, could be due to two reasons: work softening and the porosity of the material. The work softening phenomenon in shock loaded metals has been observed by Meyers [13]. As proposed by Meyers [13], work softening is due to structural rearrangements occurring during the plastic deformation.

The porosity of the material affects its strength by reducing the effective cross-sectional area under load. The densities of the consolidated powders can be achieved easily above 90 percent of theoretical density, and therefore, porosity accounted for less than 10 percent. In order for explosive compaction to ever be feasible as a powder consolidation technique, all interparticle gaps (porosity) have to be eliminated. Even 1 percent porosity will cause damage if it occurs as very thin gaps between particles as was observed even in the best consolidated material.

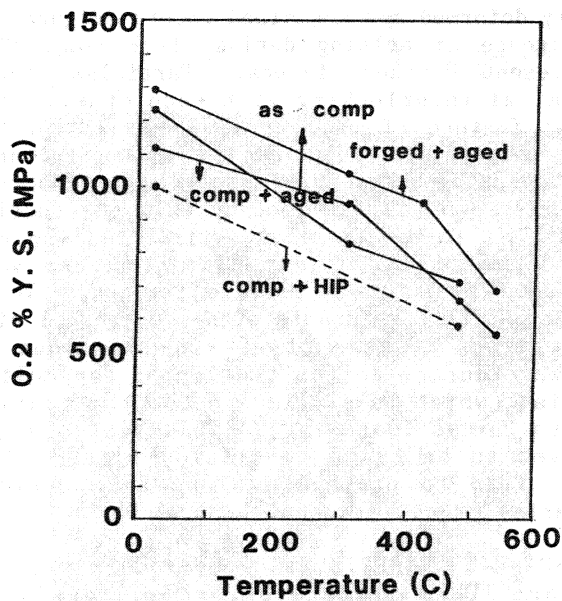
Some of the fracture surfaces of poorly consolidated specimens (broken easily by hand), and the failed tensile samples were observed with a scanning electron microscope. The fracture surfaces of the sample (broken by hand)

showed that the individual particle surfaces were heavily deformed and contained facets (Figure 8). No evidence of melting during shock compaction was present in these facets. Particles showed mechanical interlocking with each other. The typical feature of the surface observed in the well compacted specimens is the dimple structure, which is characteristic of the ductile fracture mode, Figure 9. It is thought that the ductile material is formed by interparticle melting or recrystallization. Voids and microcracks were also observed in these ductile regions. Voids are thought to form due to either entrapped air, which exists because of no vacuum or due to shrinkage during solidification of the molten material (which is similar to the the tearing defects in cast material). These defects are believed to be crack initiation sites. The cracks tend to propagate along the particle boundaries in an interparticle mode.

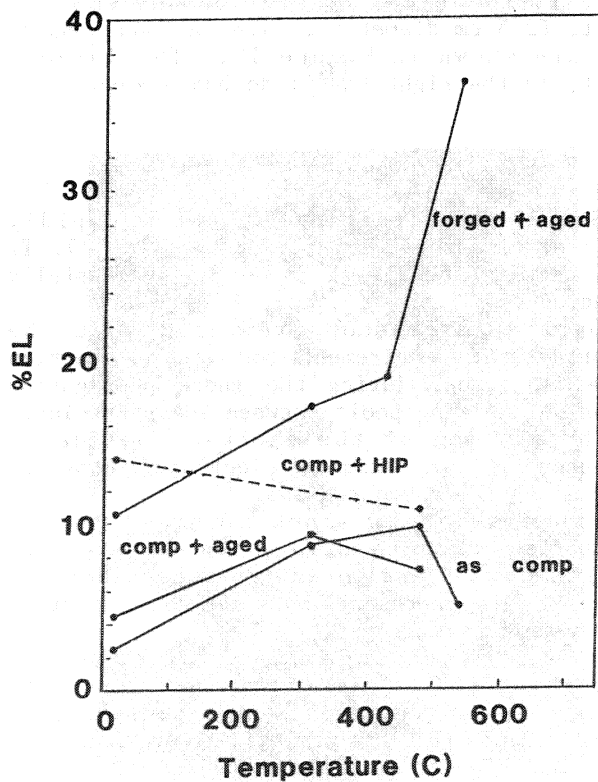
**SCALE-UP EXPERIMENTS** - Cylinders with 5, 7.5, and 10 cm diameters were successively consolidated by scaling up the consolidation fixture of Figure 1(b) while maintaining a constant initial shock pressure. The microstructures of the scaled-up compacts were virtually identical to that of the smaller compacts (2.5 cm diameter). The various compact sizes are shown in Figure 10. The largest compact, in the right hand side had a mass of 10 kg.

## CONCLUSIONS

1. Shock consolidation of three rapidly solidified titanium alloy powders -- Ti - 17, Ti - 662, and Ti - 6242 + 1% Er -- was successfully carried out.
2. Optical microscopy revealed that the microstructural requirement for good compaction is the formation, during the shock propagation process, of melting pools between the particles.
3. Observation of the shock-consolidated structures by transmission electron microscopy showed essentially two regions.
  - a) a severely shock-affected region characterized by high dislocation density (for Ti-17) and twins and laths (for the other two alloys). This represents the typical structure of the particle interiors.
  - b) a region consisting of equiaxed micrograins with low dislocation density in the interiors. This represents the interparticle regions, in which recrystallization and/or melting and rapid resolidification has occurred.
4. Shock consolidation of the titanium alloy containing erbia did not result in the exaggerated growth of these dispersoids; thus, the high-temperature strengthening phase is retained.
5. The yield and tensile strengths of shock consolidated and shock consolidated + aged alloys are of the same order as those of forged and cast alloys. The ductility is, however, considerably lower, and it does not show any marked increase



(a)



(b)

Fig. 7 - Variation of (a) yield stress and (b) total elongation with temperature for Ti - 662 in shock-consolidated, shock-consolidated + aged (570°C/4 hours), shock-consolidated + HIPped conditions; forged + aged (570°C/4 hours) material given as comparison.

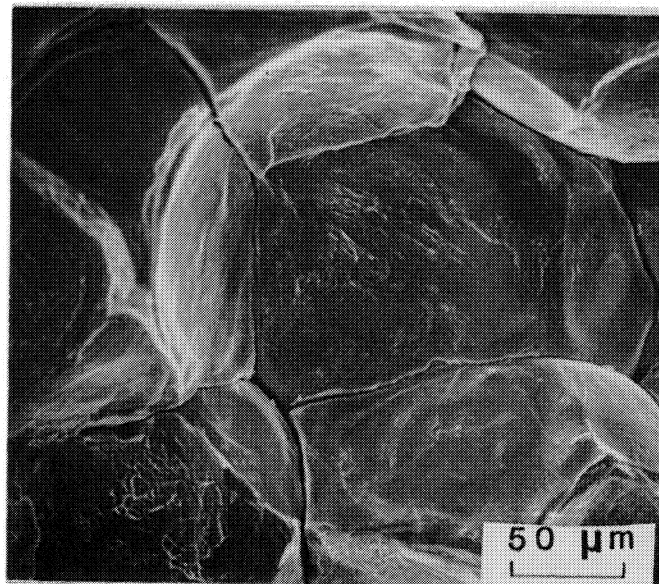


Fig. 8 - Scanning electron fractograph of poorly shock-consolidated material; notice easy separation at particle interfaces.

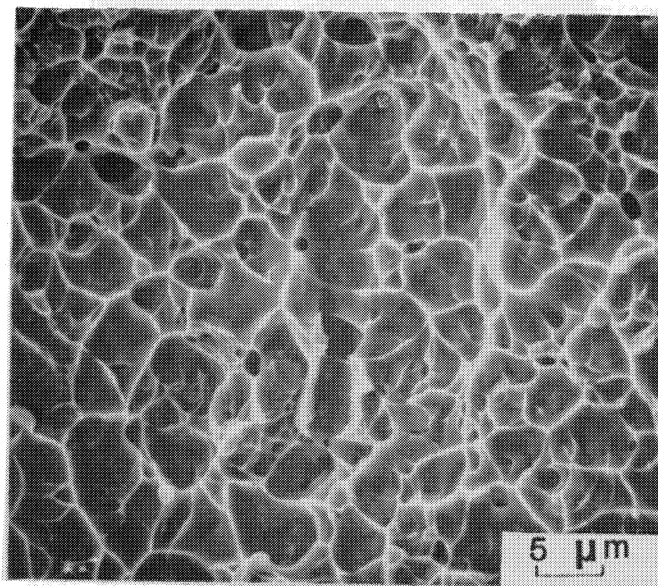


Fig. 9 - Scanning electron fractograph of well consolidated material; the dimple morphology is characteristic of the ductile fracture mode.

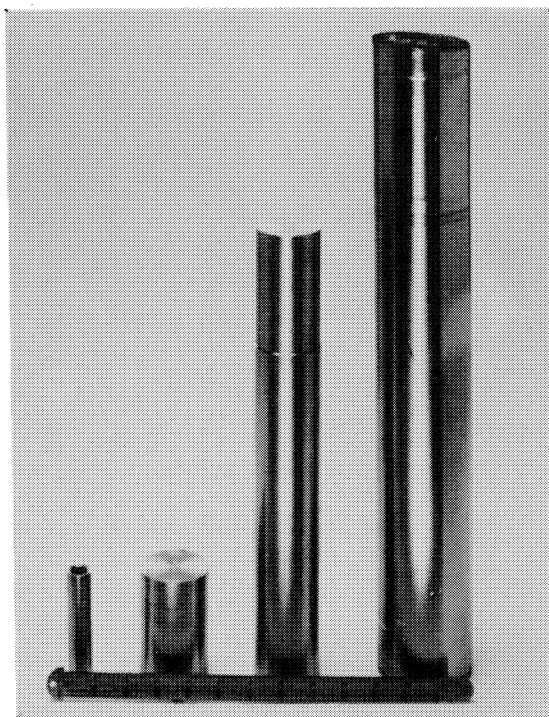


Fig. 10 - Shock compacted cylinders of different sizes showing that scaling up in size can be achieved.

at higher temperature. Hipping the shock-consolidated titanium alloy Ti-662 considerably increases the total elongation.

6. The process of shock consolidation does seem to lend itself to scale-up without difficulty.

#### ACKNOWLEDGEMENTS

This research was supported by General Electric Company and by the Center for Explosives Technology Research. Special gratitude is extended to the staff of TERA for performing the shock consolidation experiments and to Mr. A. Johnson, Manager of Advanced Metallic Materials, GE, for providing funding for this program. The authors would like to gratefully thank Dr. G. Scott and Mr. J. Wessels for their excellent transmission electron microscopy.

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