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SHOCK CONSOLIDATION OF IN-100

NICKEL-BASE SUPERALLOY POWDER

S.L. Wang, M.A. Meyers and R.A. Graham\*

Center for Explosives Technology Research, New Mexico Institute of Minning and Technology, Socorro, NM 87801 \*Sandia National Laboratories, Albuquerue, NM 87185

### INTRODUCTION

Shock consolidation is a technique of some potential in producing bulk parts from rapidly-solidified powders 1-3. Although successful reports of shock consolidation of several rapidly-solidified powders have been made, the shock-compression conditions needed are typically not well known. The objectives of the experiments described herein were to establish the stress, stress duration, and temperature conditions required for consolidation of a nickel-base superalloy. In order to accomplish this, the fixtures developed by Graham, Webb, and Davison 4,5 at Sandia National Laboratories were used. These fixtures were developed to produce quantitative, reproduceable experiments with well-characterized stress and temperature histories, determined by extensive studies with a two-dimensional code.

The alloy used in this investigation was a nickel-base superalloy powder prepared in powder form by the rotating-electrode process. The powders were spherical and had a mean diameter of 74  $\mu$ m. The nominal composition of this IN-100 powder is (in wt. pct.): Ni - 60; Cu - 10; Co  $\dot{=}$  15: Mo - 3; Al - 5.5; Ti - 4.7; C - 0.18; B - 0.014; Zr - 0.06. These powders exhibit a high strength and have been proven to be difficult to consolidate. The tensile yield stress of wrought IN-100 (bulk) exceeds 1 GPa.

The shock consolidation fixtures used are listed in Table 1. The stresses and stress durations vary throughout the capsules and the range is shown. The average stress and stress duraction can be considered as figures of merit, for comparison purposes between the various fixtures. It can be seen that both the pressure and stress duration were varied in the present experiments. The powders were kept under an argon atmosphere prior, during, and immediately following the experiments.

# RESULTS AND DISCUSSION

The degree of consolidation was not uniform throughout the capsules. It is generally accepted for metals that consolidation requires interparticle melting, while densification can be achieved in the solid state. These differences are illustrated in Figure 1.

Table 1. Shock Consolidation Fixtures Used

Fixture	Explosive	Pressure, GPa		Duration
		Range	Average	ha
Baby Bear	Baratol	13-26	20	2.5
Baby Bear	Comp B	22-32	27	2.6
Momma Bear	Baratol	5-10	7.5	4.5
Momma Bear A	a Bear A Baratol a Bear A Comp B	14-20	16	4.5
Momma Bear A		19-26	22	4.7
		14-26	17	9.8

Figure 1 shows the microstructure of the powder consolidated at an average stress of 22 GPa and duration of 4.7 µs (Momma Bear A Comp. B). Figure 1(a) shows the densified material. Notice that the particles are deformed, filling most of the voids. This material exhibits, however, very low strength. The white regions in Figure 1(b) are thought to be produced by interparticle melting which is an important consolidation parameter. Meyers et al.<sup>2</sup> showed, for Mar M 200, that the white regions were microcrystalline. It is generally thought that melting occurred, although crystallization cannot be discarded.

In Figure 1(c) hardness indentations made in the center of the particles (where a microdendritic structure may be observed) and in the interparticle melting regions can be seen. The hardness in the interior of the particles was found to be higher than in the white regions. The small indentations gave a hardness of HVN 646, while the larger indentation gave a hardness of HVN 451. Figure 1(d) is a scanning electron micrograph showing a fracture in a region where compaction occurred, but no "interparticle melting" was evident. The particles are not welded together, but simply densified. Comparing Figures 1(a) and 1(b), one can see that 1(b) shows more apparent interparticle melting.

Another interesting aspect of the consolidated material is that very often a void is observed in the center of the interparticle melt region. These voids are thought to be formed by contraction during solidification of the molten material, and are potential weakening elements, acting as nucleation sites for cracking. Figure 2 shows such a void, in the center of a white region. Another possibility for the formation of these voids is the entrapped gases. The voids between the particles were filled initially with argon.

Figure 3 shows photographs of cross-sections cut through the various fixtures. The black indicates material which had enough integrity not to flow out of capsules when they were opened and can be called "compacted" material. The three systems were drawn in different scales, so that they appear to have identical dimensions; this was done to help the comparison. The different series have different dimensions; the volumes of the capsules are 1, 5, and 60 cm<sup>3</sup> for the Baby Bear, Momma Bear, and Bertha systems, respectively. Momma Bear Baratol did not yield any compaction. It is evident from Figure 3 that there is a preference for compaction along the edges, although the pressure is somewhat lower at the edges. The explanation for this is given in Figure 4, where the temperature is plotted as a function of pressure at both the center and the edge of the sample. At the edge it is known from numerical simulations and experiments that the temperature is higher<sup>6</sup>. Thus, one can conclude that the shock temperature is an important parameter.

Figure 5 shows that the amount of interparticle (apparent) melting and therefore, the quality of the consolidate, depends on the temperature. Interparticle (apparent) melting was measured by the point counting tech-

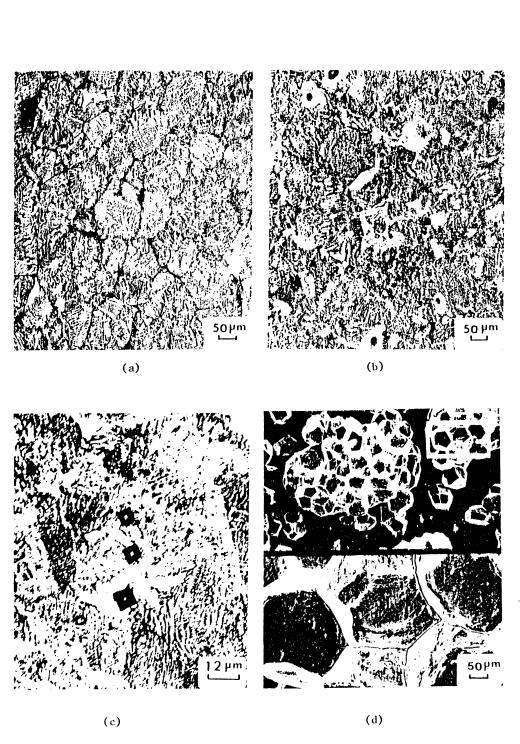


Fig. 1. Micrographs of compacted samples from Momma Bear A, Comp. B;
(a) Optical micrograph of center; (b) Optical micrograph of edge; (c) Microindentations in particle center and apparent melt regions; (d) Scanning electron micrographs of a fracture.



Fig. 2. White interparticle region with void at center.



Fig. 3. Compacted sections from different fixtures, no consolidation in Momma Bear Baratol.

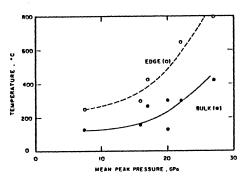


Fig. 4. Calculated maximum temp. as a function of peak pressure for various systems at edge and in center<sup>6</sup>.

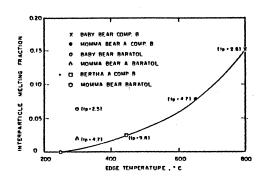


Fig. 5. Interparticle melting fracture as a function of edge temperature for the different systems. In the figure tp is the stress pulse duration.

nique applied to photomicrographs. As the temperature is increased, the fraction of interparticle melting increases. Figure 6 shows that the pressure at the edge is also important; hence, it is shown that both pressure and temperature are important.

Table 2 shows the fractions compacted for the various systems. The fractions compacted are the ratios between the black areas in Figure 3 and the total rectangular areas. It can be seen that the pressure is an important parameter. There is also an increase in fraction compacted with pulse duration, at a constant (average) pressure. For 16-20 GPa, the fractions compacted are 0.27, 0.48, and 0.64, for average durations of 2.5, 4.5, and 9.8  $\mu s$ , respectively. The interparticle melting fraction, however, did not show any significant dependence on pulse duration, at a constant pressure.

From the above observations, one can infer that compaction takes place primarily at the shock front and that an increase in pulse duration, from 2.5 to 9.8  $\mu s$ , does not result in a systematic increase in the fraction consolidated.

rraction Compacted	for the various	rixtures
Average	Average	Fraction
Pressure	Pulse	Compacted
GPa	Duration μs	
20	2.5	0.27
27	2.6	0.68
7。5	4.5	0
1 16	4.5	0.48
1. 22	4.7	0.78
17	9.8	0.64
	Average Pressure GPa  20 27 7.5 1 16 1. 22	Pressure Pulse Duration µs  20 2.5 27 2.6 7.5 4.5 1 16 4.5 1 22 4.7

The effect of temperature can be qualitatively described by Figure 7. In a shock front, the energy irreversibly dissipated is  $\Delta E_1$ , (Fig. 7(a)). If the pressure reaches its maximum after the material has been densified by a lower stress wave, the temperature increase is much lower. In a shock front, the jump conditions establish the irreversible work, most of which manifests itself as a temperature increase; a small fraction is stored as defects in the material. A gradual increase in pressure, on the other hand, allows densification at lower pressures, with less energy release. If the high-pressure pulse travels in a material that is already densified, the residual temperature increase is much less significant.

### SUMMARY AND CONCLUSIONS

Shock consolidation of IN-100 powders was studied in Sandia Bear and Bertha fixtures over a pressure from 7.5 to 27 GPa. In addition, the influence of pulse duration from 4.5 to 10  $\mu$ s was investigated at 16 GPa. From the metallographic observations it was established that:

- a) the system subjected to a 7.5 GPa average pressure did not exhibit any consolidation. From Figure 6, it can be seen that a local pressure of approximately 20 GPa is required to produce a 10 pct interparticle melting fraction. Microindentation hardness tests showed that interparticle melting is required to assure bonding between the particles.
- b) while the fraction compacted increases with increasing pulse duration, at a constant pressure, the apparent melting fraction does not show a systematic variation with pulse duration.

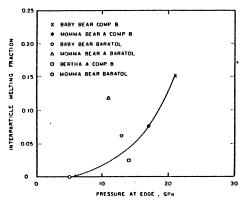


Fig. 6. Interparticle melting fraction as a function of pressure at edge.

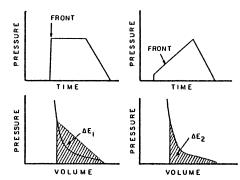


Fig. 7. Irreversible energies (heat)  $\Delta E_1$ , and  $\Delta E_2$ , for different stress-wave configurations; (a) rapidly rising stress;

(b) slowly rising stress.

- c) the shock temperature is an important parameter. It significantly enhances interparticle melting. At identical peak pressures and pulse durations, the regions undergoing the highest temperatures exhibit the greatest amount of (apparent) interparticle melting.
- d) in order to specify the requirements for a good shock consolidation, the two most important parameters that have to be specified are peak pressure and peak temperature.

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