SHOCK CONSOLIDATION OF POWDERS - THEORY AND EXPERIMENT

T. VREELAND, JR., P. KASIRAJ, AND THOMAS J. AHRENS,
California Institute of Technology, Pasadena, CA 91125
AND R. B. SCHWARZ, MST Division, Argonne National Laboratory,
Argonne, IL 60439

ABSTRACT

A recently proposed model of shock consolidation of powders quantitatively predicts regimes of input energy and shock duration required to produce well-bonded compacts. A growing data base from shock experiments in which the shock wave and powder parameters of importance are controlled allows evaluation of the model.

Rapidly solidified crystalline AISI 9310, and microcrystalline Markomet 3.11, as well as amorphous Markomet 1064 and crystalline Mo powders, have been consolidated by shocks up to 2 μsec duration. The formation of amorphous layers on Marko 3.11 particle surfaces indicates that surface melting and rapid solidification occurred. Decreasing amounts of amorphous structure are retained in Marko 3.11 and 1064 powder compacts with increasing shock energies. Significant improvement in Mo particle bonding is achieved by reducing surface oxides prior to shock consolidation.

INTRODUCTION

Relevant parameters in a recently reported model for the shock consolidation process are a dimensionless shock energy, ε(shock energy divided by the energy required to heat solid powder to its melting point), and a dimensionless shock duration, τ(shock duration divided by the time constant for a powder particle with hot boundary and cool interior to reach a homogeneous temperature)[1]. The condition that the shock energy must be sufficiently large to produce densification and melt for good bonding places a lower limit on ε. The condition that the liquid must solidify and gain some strength while under a compressive stress places a lower limit on τ($\tau \propto \varepsilon^2$ for $\varepsilon \ll 1$ and $\tau \to \infty$ as $\varepsilon \to 1$). Once the thermodynamic data for a given powder is known, the model can be used to determine approximate regions in ε and τ space which should produce good interparticle bonding. Conditions for good consolidation can be conveniently expressed in terms of shock consolidation maps which plot τ vs ε.

It has been experimentally demonstrated that some minimum energy is required to produce densification and sufficient interparticle melting for good bonding [2,3,4]. Intense localized plastic deformation near particle boundaries and interparticle friction convert most of the shock energy into heat during the shock rise time. Morris [4] and Gourdin [5] have presented metallurgical evidence of the inhomogeneous nature of the energy deposition which can give rise to particle surface melting. Measurement of the tensile properties of consolidated powders provides a quantitative test of interparticle bonding, while observations on fractured samples provide qualitative information.

The shock energy may be approximated by $E = P_1 V_0 (m - 1)/2$ where P_1 is the pressure in the initial shock which densifies the powder, mV_0 is the specific volume of the powder (m = distention), and V_0 is the specific vol-

Mat. Res. Soc. Symp. Proc. Vol. 28 (1984) Published by Elsevier Science Publishing Co., Inc.

ume of the solid. A shock consolidation map for AISI 9310 steel powder has been previously reported [1]. The present shock recovery experiments on powders of Marko 3.11 and 1064 alloys and AMAX molybdenum provide additional information relevant to consolidation maps for these powders.

EXPERIMENTAL METHODS

Rapidly solidified powders produced from ribbons of Marko 3.11 and 1064 are shown in the SEM micrographs of Figs. 1 a,b. The AMAX Mo powder is shown in Fig. 1 c. The powders were statically compressed into green compacts of controlled distention which were impacted by flyer plates to generate plane shock waves. The duration of the shock was approximately 2 μs, and the powders were evacuated to 40 μm Hg before shocking at 22°C.

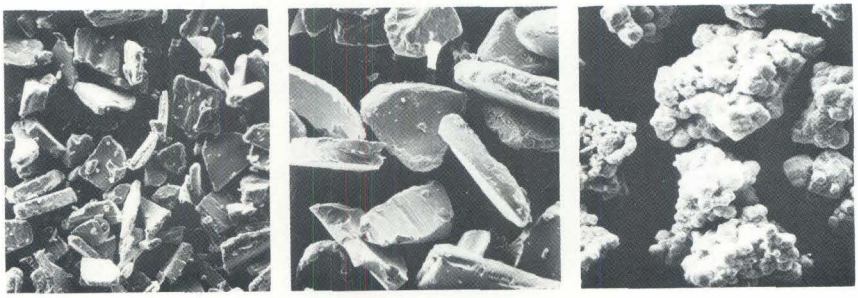

FIG. 1. SEM micrographs of powders (a) Marko 3.11, (b) Marko 1064, (c) AMAX Molybdenum.

The powders and recovered powder compacts were mounted in lucite for metallographic examination and microhardness measurements. X-ray diffraction scans were made of the Marko powders and some of the Marko compacts and small samples were spark machined from the molybdenum compact for tensile tests.

TEST RESULTS AND DISCUSSION

Marko 3.11 ($Fe_{Bal}W_{5.75}Mo_{4.5}Cr_{4.25}V_{4.0}C_{1.38}B_{0.65}$, wt.%)

The Marko 3.11 powders (-400 mesh, <38 μm) were observed to have a thin layer of non-etching amorphous phase on one side (presumably the side which contacted the wheel in the melt spinning process), with the remainder microcrystalline, Fig. 2 a. Photomicrographs of etched cross sections of compacts produced at three different shock energies are shown in Fig. 2 b-d. Table I gives the experimental parameters together with a summary of the microstructural observations. The thermal parameters used to obtain the ε values and $\tau = 1.7$ were $\bar{c}_p \Delta T_m = 911$ kJ/kg, $D_m = 5.5 \times 10^{-6} m^2/s$ (notation of Ref. 1). The microcrystalline phase was BCC (a = 2.88 Å) and a small fraction of an FCC phase (a = 3.58 Å) was observed in the compact of Fig. 2 d.

As the shock energy increases the amount of amorphous phase is observed to initially increase in the mixed amorphous-microcrystalline particles, and then decrease. This indicates shock induced formation of a liquid phase which is rapidly quenched at the lower shock energies, while shock heating and slower cooling transform some of the amorphous phase to microcrystalline

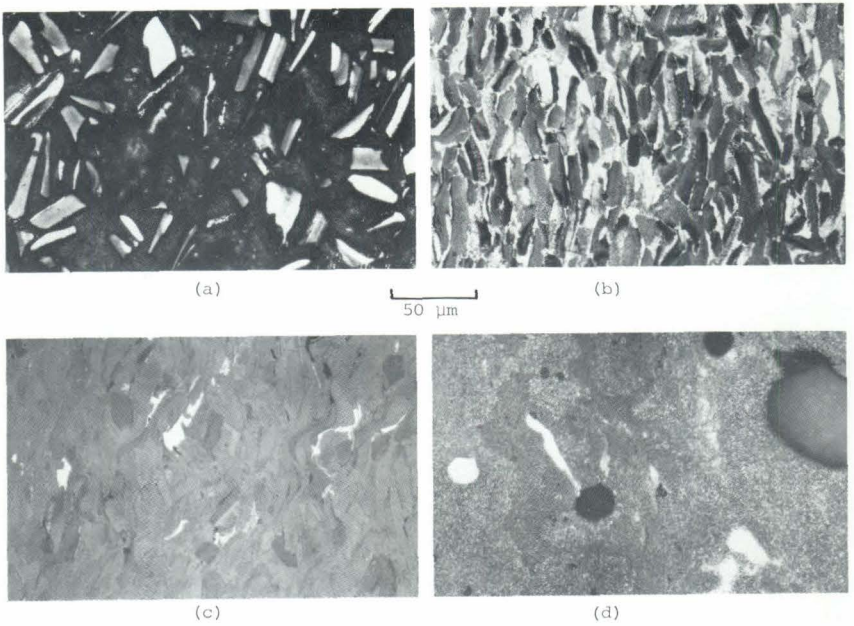

FIG. 2. Optical micrographs of (a) Marko 3.11 powder, (b)-(d) sections of compacts, shock propagation left to right and shock energy increasing from (b) to (d) (see Table I). Etch: Marble's reagent, 5 s.

TABLE I. Marko 3.11 Shock Consolidation (a = amorphous, µx = microcrystal)

Fig.	m	P_1(GPa)	E(kJ/kg)	ε	DPH (500g)	Structure	Particle Fracture
2 b	2.09	5.2	345	0.38	1190	mixed a, µx	intra + inter
2 c	2.09	7.2	477	0.52	1131	mostly µx	mostly intra
2 d	2.09	13.8	915	1.00	925	mostly µx + bubbles	———

material at higher shock energies.

Marko 1064 ($Ni_{52.5}Mo_{38}Cr_8B_{1.5}$, wt.%)

Marko 1064 is initially amorphous. Differential thermal analysis, microstructural observations of polished and etched powder before and after thermal treatments, and X-ray diffractometry indicate an amorphous to microcrystalline transformation occurs near 600°C. Photomicrographs of etched sections of compacts produced at three different shock energies are shown in Fig. 3 (a)-(c). The amorphous phase shows no discernible etching in 20 min. Table II gives the experimental parameters together with a summary of microstructural observations. The thermal parameters used to obtain the ε values and $\tau = 0.43$ are $\bar{c}_p\Delta T_m = 520$ kJ/kg and $D_m = 1.33 \times 10^{-6} m^2/s$. Initially the particles have a DPH (50g) = 987±156 kg/mm^2.

At the lowest shock energy only isolated particles or portions of particles show transformation to the microcrystalline phase, in amounts too small

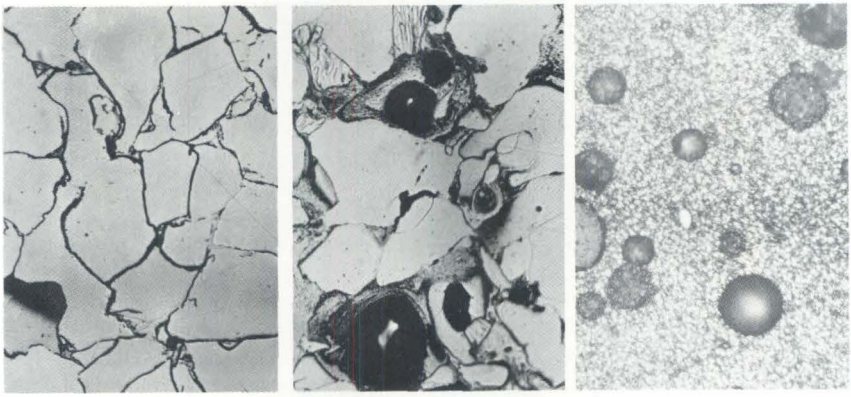

FIG. 3. Optical micrographs of Marko 1064 compacts, viewed in the plane of the shock front, shock energy increasing from (a) to (c) (see Table II). Etch: Marble's reagent, 20 min.

TABLE II. Marko 1064 Shock Consolidation (a = amorphous, μx = microcrystal)

Fig.	m	P_1 (GPa)	E(kJ/kg)	ε	DPH (50g)	Structure	Particle Fracture
3 a	2.03	4.0	230	0.44	1330	a + few μx regions	inter
3 b	2.00	7.7	430	0.83	1200	a + μx, few bubbles	intra
3 c	1.94	12.0	630	1.22	652	μx + few a, bubbles	____

to detect in diffractometer scans. The amount of material transformed to the microcrystalline phase increases with increasing shock energy, and the highest shock energy produced a mostly microcrystalline compact with many macro bubbles. Softening at the highest shock energy may be attributed to grain growth and possibly to coarsening of boride percipitates.

AMAX Molybdenum (99.8 wt.% Mo)

Shock energies in the range 185 to 894 kJ/kg failed to produce well-bonded compacts of the as-received powder. An X-ray diffractometer scan of recovered fragments of a compact indicated the presence of tetragonal MoO_2 and suggested a possible problem with surface oxides and oxidation during the consolidation. A photomicrograph of the powder is shown in Fig. 4 a. The powder was treated at 750°C in an atmosphere of H_2 (dew point ~60°C) for 4 h and loaded into a target assembly under an Ar atmosphere. After a 1 minute exposure to air, the target assembly was filled with Ar and the pressure reduced to 40 μm Hg. A shock of E = 580 kJ/kg produced a compact with the microstructure shown in Fig. 4 b (m = 1.67, P_1 = 17.8 GPa). The hardness of the compact was measured to be comparable to that of the powder (DPH(5g) = 249). Tensile tests on the consolidated Mo gave a UTS of 0.76 GPa (110 ksi) which is comparable to rolled sheet or bar stock. Using $\bar{c}_p \Delta T_m$ = 968 kJ/kg and $D_m = 1.8 \times 10^{-5} m^2/s$ we obtain ε = 0.6 and τ = 0.60 for the Mo consolidation.

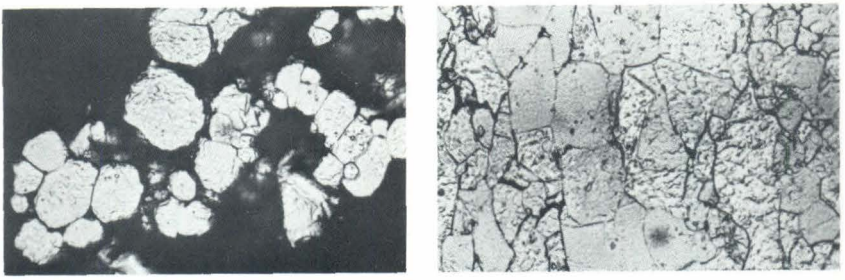

FIG. 4. Optical micrographs of AMAX molybdenum. (a) powder, (b) compact, shock propagation left to right. Etch: Murakami solution 30s (a),60s (b).

CONCLUSIONS

(1) Fully dense, well-bonded compacts of Marko 3.11, Marko 1064, and AMAX Mo powders may be produced by shock waves. The results presented here indicate optimum regions for good consolidation exist. These regions are generally consistent with predictions of the model. (2) The amount of amorphous phase in the Marko 3.11 compacts initially increases with increasing shock energy, indicating interparticle melting and rapid solidification. With further increases in energy, shock heating produces an amorphous to microcrystalline transformation in the Marko alloys. (3) Surface oxides on AMAX Mo powder particles adversely effect interparticle bonding. (4) Shock loading experiments with glass forming microcrystalline powders can clearly show the inhomogenities in energy deposition and permit the determination of particle size, shape, and size distribution effects.

ACKNOWLEDGEMENT

This work was partially supported by United Technologies Research Center and Defense Advanced Projects Agency through the U. S. Army Materials and Mechanics Research Center. Dr. B. F. Kear kindly supplied the Marko 1064 powder. Lynne K. Adler assisted in the Marko 3.11 experiments. Caltech contribution number 4005.

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