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Structure, phase content and mechanical properties of aluminium with hard particles after shock-wave compaction

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Abstract. The possibilities to combine metal and metal oxide powders in various compositions open a broad range of mechanical and thermal behavior. When using in nanostructured components the resulting materials might exhibit even more interesting properties, like product effectiveness, tensile strength, wear resistance, endurance and corrosion resistance. Intermetallics like TiAl could be obtained as $TiAl_x$ in a quality similar to that obtained from melting where only eutectic mixture can be produced. Similar effects are possible when compacting nanoceramic powders whereas these can be combined with intermetallics. Currently, it is very difficult to produce wires and special shaped parts from high temperature superconducting materials. The compacting by explosives could solve this problem. The present paper uses explosion compacting of Al nanoparticles to create nanocomposite with increased physico-mechanical properties. Russian civil explosive Uglenit was chosen as high energy material (HEM) for shock-wave compaction. The different schemes and conditions were suggested to run the explosion process. Al nanoparticles as produced by electric wire explosion contain 8-10% of aluminum oxide. That aluminum oxide can serve as strengthening material in the final nanocomposite which may be generated in various compositions by explosive compacting. Further modifications of nanocomposites were obtained when including nanodiamonds into the mixture with aluminum nanoparticles with different percentages. The addition of nanodiamonds results in a substantial strengthening effect.

The experiments with compacting aluminum nanoparticles by explosives are described in detail including the process variations and conditions. The physico-mechanical properties of the nanocomposites are determined and discussed by considering the applied conditions. Especially, microstructure and phases of the obtained nanocomposites are analyzed by X-ray diffraction.

1. Introduction

It is known [1-5], that material properties are strongly influenced by its internal structure - presence of defects, size of structural elements, presence of reinforcing elements etc. if is not always possible to form desired structure using traditional production schemes. Particularly traditional metallurgical processes are associated with increase of grain size and defect annealing, which require additional mechanical processing of an alloy. At the same time there is a method of dynamic compaction of powder mixtures with desired composition. Such techniques make it possible to obtain high degree of solidity material with density close to theoretical value; along with introduction of desired number of defects and reinforcement elements.



Such technologies are extremely important for production of Al composites by means of dynamic impact on powder aluminum alloys with necessary content of aluminum oxide as a surface film [6], which acts as a source of aluminum oxide reinforcement particles after shock loading. At the same time this method of aluminum powder compaction makes it possible to change the type and quantity of reinforcement material, e.g. aluminum oxide nanoparticles, high-modulus carbon particles in the form of nanodiamonds (further n-diamonds) [7-9] etc. This enables creation of materials with desired properties.

The objective of this work is to study properties of composites produced by explosive compaction of aluminum powder mixed with aluminum oxide nanoparticles and n-diamonds.

2. Materials and methods

2.1. Selection of explosive for Shock-Wave Compaction of aluminum powders.

It is known [6], that the pressure required for consolidation of certain material is determined by its hardness. It is pointed out that the pressure required for compaction of Al powder with regular particle size (<160 μm) and hardness Hv=100 is equal to 1 GPa (see Fig.3.13 in [10]). Some industrial explosives such as Ammonit-1, Carbonit [10], Uglenit [11] (see Table 1), do have required characteristics. Calculations performed using the formula [10] indicated that these values must be sufficient to obtain fully compacted material. Moreover, detonation rate calculation for a shock wave according to formula

$$V_d = 2 \cdot (1.2 \cdot H_v / \rho_e)^{1/2}$$

indicates that required value of detonation rate for an explosive (e.g. Uglenit) comprises 900 m/s, at the same time the value stated in [6] is significantly (more than two times) higher (see table 1). Thus, the simplest explosive "Uglenit" was selected for experiments with Al powders, providing required compaction.

$$P_H \approx 1/4 \cdot (\rho_{BB} \cdot (D_{BB})^2) [10]$$

P_H is functionally related to density and detonation rate of explosive used and according to Prummer's estimation equals to

$$P_H \approx 1/4 \cdot (\rho_{BB} \cdot (D_{BB})^2)$$

"Uglenit E6" industrial coal mine explosive with bulk density of 1.10-1.25 g/cm³ and detonation rate D=2100-2500 m/s (according to technical specifications), was used as explosive. TNT equivalent was equal to 0.45, fugacity value - 130 cm³. Estimated value of $P_H=13-15$ kBar.

Table 1. Characteristic data of some common explosives.

Explosive	ρ_e , g/cm ³	V_d , m/s	P, GPa	Ref.
Ammonit-1	1.25	3500	3.8	[6]
Carbonit	1.05	1600	0.7	[6]
Uglenit	1.2	2200	1.5	[7]

2.2. Experimental technique

A 30mm layer of explosive (pillow) compacted to $\rho=1,25$ g/cm³ was poured into a cardboard glass (Ø=40 mm). A tube was installed onto the pillow using a centering cardboard ring. The gap was also fitted with compacted explosive. A copper tube with powder mixture of designated composition was centered by means of the second ring, and then compacted explosive was put into the glass 50mm above the tube. A standard electric detonator was installed in the middle of this layer at 20 mm depth. Total mass of explosive was comprised 300g. The assembly was put into the blast chamber on a metal plate, detonator was connected to initiation grid, the chamber was dosed and the assembly was blasted. Standard industrial explosive "Uglenit E-6" was used. Design pressure in detonation wave was $P=13...15$ kBar.

Phase composition and structural parameters of initial powders and obtained materials were studied using diffractometers with CuK α - radiation. Step-by-step collection of X-ray patterns was performed

with 0.02-0.1° step in the interval $20^\circ < 2\theta < 120^\circ$. Phase identification was performed by means of comparison of experimental pattern with ASTM-data. The sizes of coherently diffracting domains (CDD) were determined by using small-angle peaks and micro-distortion of crystal lattice (ϵ) was calculated using widening of reflection at large diffraction angles [12].

The structure of the composites was examined under a Philips SEM 515 microscope. The hardness of the materials was measured by a Supervickers hardness meter under a load of 1000g. Compression tests were performed using an Instron 1185 testing machine.

2.3. Experimental material

Compaction of Al powder as well as its mixtures with n-diamond [7-9] and alumina [4, 13] (10 wt% of each) was investigated in the framework of this research. Investigation of such compositions along with practical use for production of particle-reinforced composite materials will make it possible to study influence of high-modulus components on Al compaction process. This issue still remains understudied.

3. Results and discussion

According to X-ray diffraction (XRD) analysis average crystallite size for Al powder comprised 90 nm, microdistorsion of crystal lattice – 10^{-3} . X-ray phase analysis has indicated that carbon powder consists of an X-ray amorphous phase (40%) and diamond phase (45%), the remaining part is represented by crystalline carbon. Average crystallite size for n-diamonds comprises 4 nm, microdistorsion of crystal lattice – 17×10^{-3} . According to XRD analysis average crystallite size for alumina powder comprised 30 nm, microdistorsion of crystal lattice – 5×10^{-3} .

Metallographic examinations have indicated that shock-wave (SW) treatment [14] of investigated mixtures makes it possible to achieve pore less condition (Fig. 1). The grain size of compacted materials in this case comprises 4-20 μm depending on the type of mixture, which is significantly larger than powder particle size. This appears to be the result of dynamic recrystallization which took place during explosive compaction.

It is obvious from the grain size distributions shown in Fig. 2 that the average grain size in the powders in the initial state is 220 μm and on exposure to shock waves, it reduces down to 10 μm . In this case if grain size for the samples from aluminum nanopowder comprises $20 \pm 10 \mu\text{m}$, introduction of reinforcement particles significantly reduces it down to 11 μm for n-diamond-containing mixtures, and to 4 μm for mixtures which contain aluminum oxide.

Fig.3 shows X-ray data, obtained for samples after SW-treatment of aluminum powder and aluminum-n-diamonds mixtures as well as mixtures of aluminum with nanocrystalline aluminum oxide.

Obtained data indicate that double-phase state with significantly different structural parameters was formed for samples containing n-diamond and aluminum oxide. This state is indicated with an arrow on Fig. 2 - a small reflection near the main one, which belongs to aluminum. The study of parameters of fine crystalline structure have indicated that sizes of crystalline particles of main aluminum phase in all cases are equal to $80 \pm 10 \text{ nm}$, this value is close to the size of the crystallites in the powder of aluminum in the initial state. Sizes of aluminum nanophase for SW-treatment mixtures with cluster diamonds are equal to $13 \pm 5 \text{ nm}$, while in case of aluminum oxide powder additions - they are equal to $8 \pm 5 \text{ nm}$. Lattice parameter of the nanophase is increased by 0.5% in this case, which indicates its non-equilibrium state. Such an increase may be associated with compressive stresses applied at the radius of compacted cylinder with powder mixture. Estimates have indicated that these stresses are equal to 350 MPa.

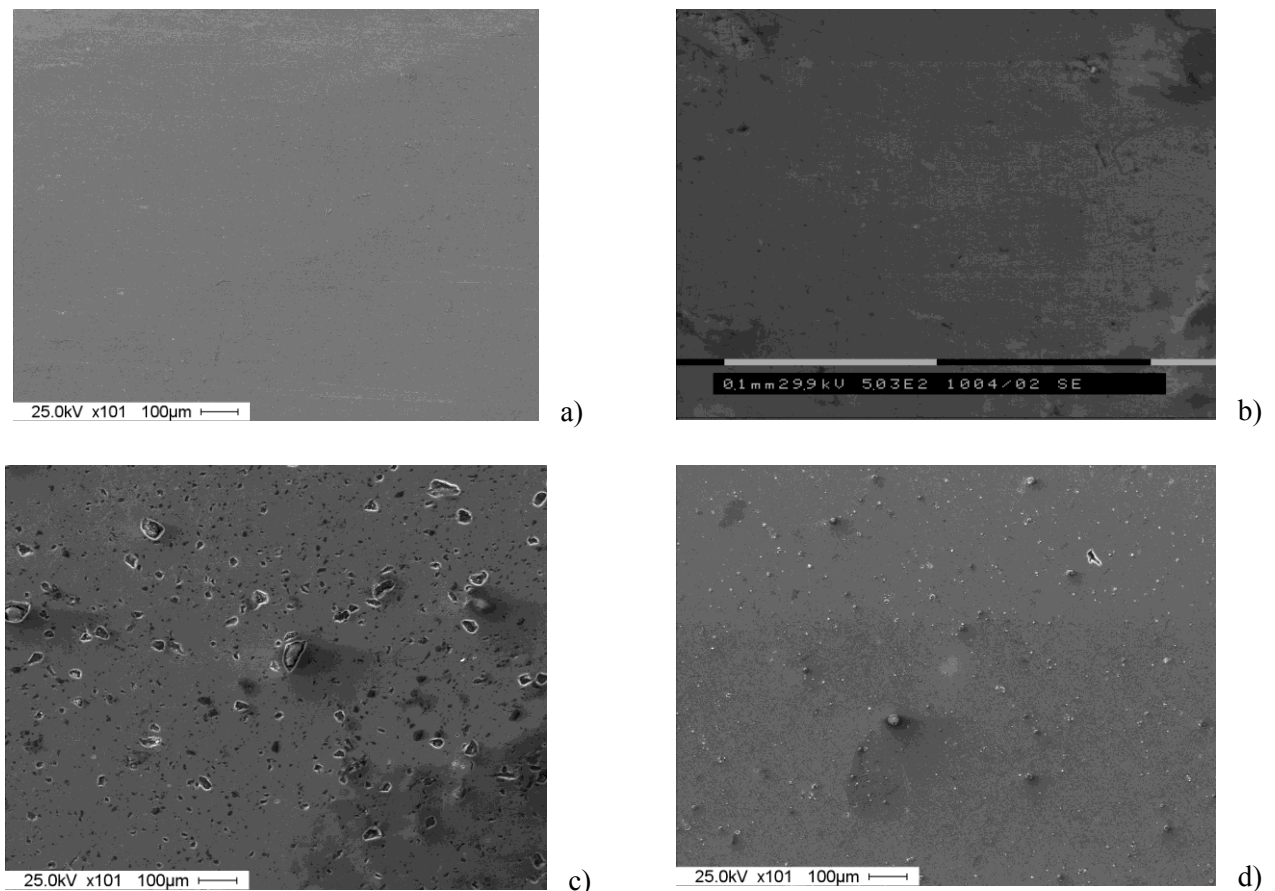


Figure 1. Microstructure of the materials: commercial 99.5 % pure aluminum (*a*) and alloys obtained by shock-wave compaction of an aluminum powder (*b*) and Al-C (*c*) and Al-Al₂O₃ (*d*).

The fact that there are many compressive stresses influences mechanical properties of SW-treated materials. Indeed, hardness measurements have indicated a 10-time increasing with maximum values for samples containing aluminum oxide. The fact that these internal stresses are determined by shock-wave treatment is confirmed by the data obtained for hardness and strength characteristics after annealing of compacted samples given in brackets in the table. As it can be seen there is a significant stress relaxation, however, it is less significant for samples with n-diamonds.

Microdistorsion of crystal lattice (Table 2) was determined using diffraction patterns for Williamson-Hall relationship. Microdistortion value has increased after adding hard particles. These values match with estimates made for metal powders [8] and make it possible to estimate stored energy [15]. As it can be seen from the table this energy increases when high-modulus particles are added. A shock wave taking place in powder containing ampoule seems to spend its energy on Al activation as well as on activation of particles. Assuming the simplest case of additive contribution to activation of Al particles by hard ones estimates of reserve energy of alumina and n-diamonds can be obtained.

The value for alumina comprised 1.1 J/kg which corresponds to data mentioned in [15], as for n-diamonds, the value was two times lower. This can be explained by complexity of phase composition of carbon mixture consisting of an X-ray amorphous phase, diamond phase and crystalline carbon.

The data regarding the change of parameters of crystal lattice of investigated materials in the process of shock-wave compaction depending on reserve energy are shown on Fig.4.

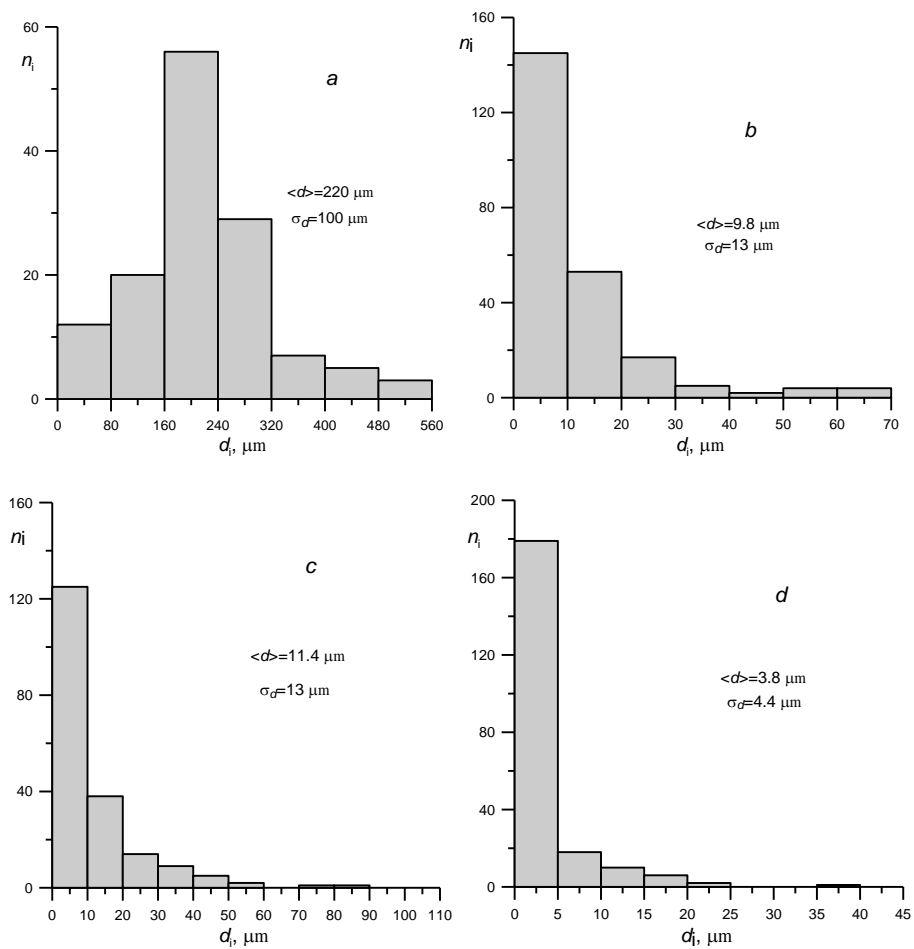


Figure 2. Grain size distribution in commercial 99.5 % pure aluminum (a) and materials obtained by shock-wave compaction of an aluminum powder (b) and Al-C (c) and Al-Al₂O₃ (d).

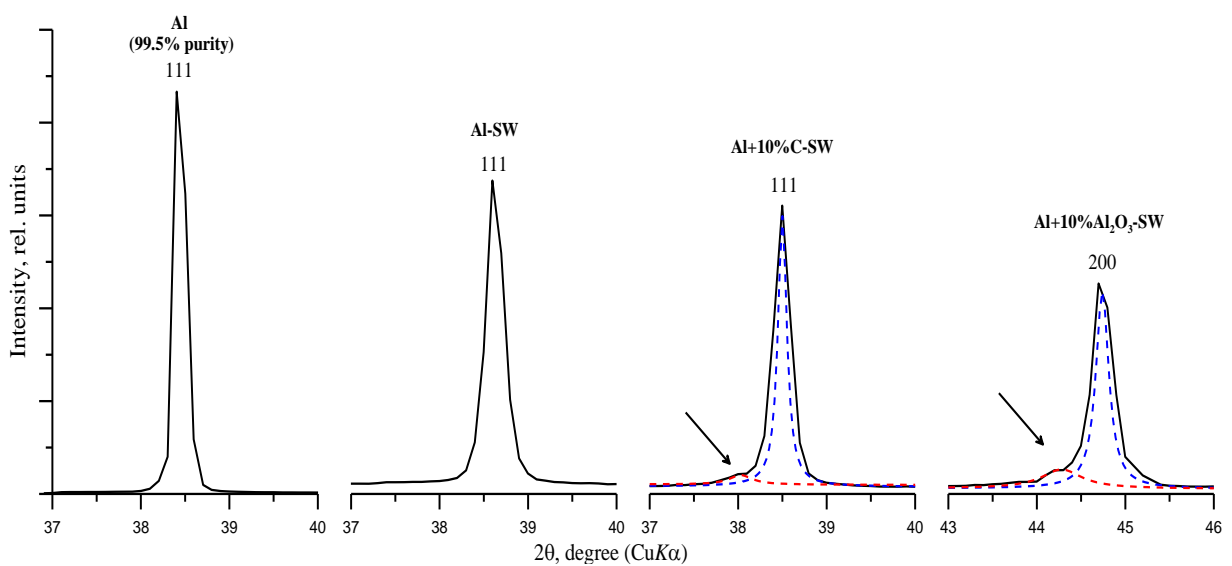


Figure 3. X-ray spectra of the samples obtained by shock-wave compaction.

Table 2. The Microstructural Parameters and Mechanical Characteristics of the Specimens

Sample	Crystallite size of aluminum /nanophase, nm	Hardness, HV, MPa	Yield strength, MPa	Strain on elastic limit, ϵ_y	Microdistortion of Al-lattice, $\cdot 10^3$	Stored energy for aluminum, J/g
Al commercial pure	155	190	30	0.0036		
Al after SW-compaction	70	870 (35 after annealing)	-	-	0.75	0.042
Al+C after SW-compaction	90 /13	1025 (190 after annealing)	400 (240 after annealing)	0.023 (0.014 after annealing)	1.16	0.099
Al+Al ₂ O ₃ , after SW-compaction	70 /8	1360 (140 after annealing)	500 (225 after annealing)	0.0205 (0.0105 after annealing)	1.43	0.151

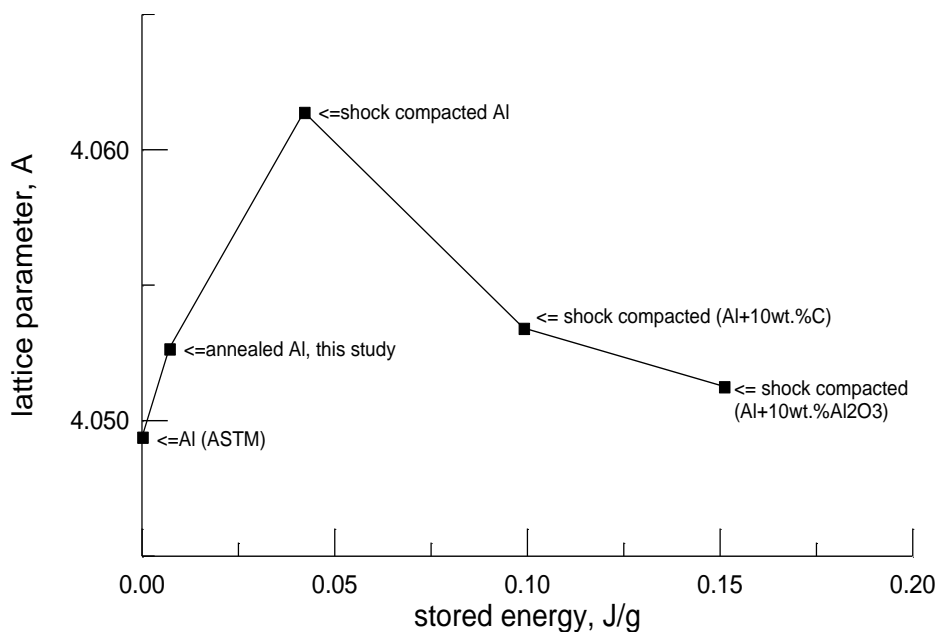


Figure 4. The change of parameters of crystal lattice of investigated materials in the process of shock-wave compaction depending on stored energy

As it can be seen this relationship has a maximum peak, which can be explained on one hand by the growth of microstrains (microdeformation of crystal lattice) and on the other hand – by annealing of defects due to more intense heating of Al powder after adding hard particles in comparison with "pure" powder.

4. Conclusion

It was shown that shock-wave treatment of aluminum powder mixtures Al+10wt.%C and Al+10 wt.% Al₂O₃ in copper capsules makes it possible to obtain samples with almost theoretical density and significantly better mechanical characteristics such as hardness and yield strength. The reason for this is that the part of material changes its state to nanostructured one with the size of structural elements equal approximately 10 nm and formation of compression stresses in a loaded samples up to 350 MPa.

Acknowledgments

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