# Formation of metal matrix composite by magnetic pulsed compaction of partially oxidized Al nanopowder

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New nanostructured AI matrix composite AI-AI<sub>2</sub>O<sub>3</sub> with homogeneous distribution of oxide particles was fabricated by magnetic pulsed compaction of partially oxidized AI nanopowders preheated up to 400–500°C in vacuum. The particles of starting powders with the mean sizes of 50–200 nm represent the grains of metal aluminum covered with the shells from hard oxide AI<sub>2</sub>O<sub>3</sub> in amorphous state. Under action of intensive pulsed pressure (1 GPa, 300 microseconds) the mass destruction of oxide shells with the formation of a metal matrix containing the reinforcing oxide nano-sized particles is realized. At pulsed compaction the simultaneous actions of high pulsed pressure, large mechanical pulse of particles and accompanying adiabatic heating are successfully combined. The sintered composite is characterized by fine homogeneous structure, full density and high microhardness (about 2 GPa), which are stable up to 400°C. The tensile strength of a material exceeds 350 GPa at room temperature. © 2004 Kluwer Academic Publishers

## 1. Introduction

The aluminum matrix composites have a set of essential advantages over conventional age hardened aluminum alloys, in particular, such as high temperature strength, good wear resistance, high Young's modulus, etc. For the parts, exploited under elevated temperatures, the aluminum alloys with thermostable mechanical properties at temperatures higher than 250°C are required. The aluminum-based composite strengthened by inclusions of own oxide Al<sub>2</sub>O<sub>3</sub> is the perspective candidate for such applications. Among approaches used earlier for fabricating of a composite Al-Al<sub>2</sub>O<sub>3</sub>, in particular, the following are known: mechanical alloying with the subsequent static compaction [1], and static compaction of partly oxidized (passivated) nanopowders [2]. In the latter case, the authors did not manage to get full density and high mechanical properties of a material. The probable reason could be insufficient efficiency of the applied static compaction, which has not allowed realizing global destruction of oxide shells of aluminum particles. In our previous work [3], we proposed to use magnetic pulsed compaction (MPC) for effective formation of a composite from passivated aluminum nanopowder. In the present paper, the results of the study of compaction dynamics, structure and mechanical properties of sintered metal-matrix composite (MMC) Al-Al<sub>2</sub>O<sub>3</sub>, are presented.

#### 2. Experimental

The aluminum-based nanopowders used were produced by electrical explosion of aluminum wires, in the Institute of Electrophysics, UD RAS, Ekaterinburg. After synthesis in an neutral gas atmosphere, the metal powder have been passivated by slow air filling. According to the data of the volumetric analysis, transparent electron microscopy (TEM) and X-ray analysis the powders contained about 2–15 wt% of aluminum oxide in amorphous state, mainly as a thin shell (2–4 nm) covering the metal grains.

The uniaxial MPC of powders in cylindrical mould was used to obtain disks (32 mm in diameter and 1-2 mm thick). The amplitude and duration of compacting pressure pulses were 1 GPa and 300 microseconds, respectively. Prior to compaction, the powder was degassed in vacuum under residual pressure of  $\sim 1$  Pa at the temperature from the range 400–500°C for 2 h. Compaction was carried out at degassing temperatures under uninterrupted vacuum pumping. The MPC installation developed allows to conduct all processing steps from powder packing into a mould to pressing out a compact inside the unified vacuum system. The important feature of the pulsed compaction is adiabatic character of the process resulting in an additional pulsed heating of a powder to hundreds of degrees. This effect was investigated using adiabatic compression method

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#### MECHANOCHEMISTRY AND MECHANICAL ALLOYING 2003

[4] that provides the plotting of dynamic compression adiabatics  $\gamma(P)$  and determining a compression work w(P) as a function of pressure *P*. After densification, the compacts were exposed to additional annealing in the temperature range of 400–525°C, providing a removal of internal mechanical stresses.

The X-ray phase and structural analysis of starting powders and compacted samples was executed using the device DRON-4M with Cu-K<sub> $\alpha$ </sub> radiation. The mean size of aluminum crystals was determined using the Sherrer method. The microhardness was measured using the PMT-3 device under 40 g loading with an accuracy of 10%. Density of samples was measured by a standard Archimedes method. The samples for measurements of tensile strength in the shape of bar with 2 × 0.4 mm<sup>2</sup> in cross section were prepared by machining from compacted discs. The strength measurements were made in the temperature range of 20– 400°C. For structure investigations, the TEM, model Jeol 200, and atomic force microscope Solver 47 were applied.

#### 3. Results and discussion

The processes of pulse pressing are characterized by severe constraints, at which it is possible to expect intensive destruction of oxide shells around the starting particles of a passivated aluminum powder. The first evidence for that was taken from the data on compression dynamics at MPC of powder under investigation. For three compaction regimes with various pressure pulses of 0.30, 0.52 and 0.95 GPa in amplitude, the adiabatic compression curves,  $\gamma(P)$ , and compression work functions, w(P), are shown on Fig. 1. The increase of dynamic compressibility with pressure growth at the final compression stage can be seen from the shape of  $\gamma(P)$  curves (Fig. 1a). Such behaviour is unusual for near full dense compacts at conventional static compactions. Furthermore, in the cases of 1 and 2 compaction modes the slope of curves  $\gamma(P)$  became negative values at the maximum pressures. That means, formally, negative compressibility of dynamically compacted powder. The found effect can be explained with essential adiabatic heating of powder during MPC compression process. According to estimation, using the measured compression work w(P) (Fig. 1b) under action of pulsed pressure of 1 GPa, an additional adiabatic heating up of an aluminum powder to 200°C is possible. Here the heat capacity of aluminum was considered to be equal 25 J/mol · K. In view of preliminary preheating to 400–500°C, the total pulsed temperature of compacted powder at final compression stage should reach the melting point of metal aluminum.

Microscopic investigations reveal near full densities and homogeneous nano-scale structure of compacts for all investigated compaction modes. At the same time, the essential structure peculiarities were found between samples compacted at various temperatures. For compacts prepared at the room temperature, the aluminum grains close to the spherical shape, and also Al<sub>2</sub>O<sub>3</sub> particles in the form of thin plates (see dark zones) located on grain boundaries are visible (Fig. 2a). On the contrary, in the samples compacted at 400°C and at the higher



*Figure 1* Compression adiabatics,  $\gamma(P)$ , (a) and compression work function, w(P), (b) of passivated aluminum nanopowder compacted dynamically at room temperature at three MPC regimes characterized with various pressure pulses of 0.30 (curve 1), 0.52 (curve 2) and 0.95 GPa (curve 3) in amplitude.

temperatures there are dominating areas, where the oxide covers are completely destroyed and small platelike alumina particles uniformly distributed inside the metal matrix (Fig. 2b). Sometimes, in such samples the separate metal crystals do not distinct. On electron diffraction patterns from different samples the dotted diffraction rings were observed showing the presence of only aluminum in nano-crystalline state and the diffuse halo testifying about amorphous alumina. The structural data are indicative for formation at pulsed hot compactions (>400  $^{\circ}$ C) of the composite with the aluminum matrix and strengthening particles of amorphous Al<sub>2</sub>O<sub>3</sub>. In such process, the mass destruction of oxide shells around aluminum grains with formation of a metal matrix containing the oxide particles is realized.

The sintered nano-scale composite Al-Al<sub>2</sub>O<sub>3</sub> possesses full density and high microhardness, about 2 GPa, which are stable up to temperature of 400°C. Fig. 3 demonstrates the dependence of tensile strength vs. temperature of tests for the samples pressed and annealed at temperatures 450–500°C. The dotted curve fits the highest values of tensile strength at different temperatures characterizing the best realized mode of compaction and heat treatment. As a positive effect in the behavior of strength it is necessary to recognize its weak fall with temperature growth up to 350°C. It well correlates with thermal stability of a composite

### **MECHANOCHEMISTRY AND MECHANICAL ALLOYING 2003**



(a)



(b)

*Figure 2* Typical structure of Al-Al<sub>2</sub>O<sub>3</sub> composites sintered by pulsed compaction of passivated aluminum nanopowder at starting temperatures of  $20^{\circ}$ C (a) and  $400^{\circ}$ C (b).



*Figure 3* Dependence of tensile strength vs. temperature of test for composites compacted and annealed at temperatures of  $450-500^{\circ}$ C.

structure. The given property favorably distinguishes such material from conventional agehardened aluminum alloys, which are characterized by low temperature stability [5]. The significant scattering of the data for a series of samples made under the close conditions can be connected with the sensitivity to variations in technological regime.

### 4. Conclusion

Nanostructured metal-matrix  $Al-Al_2O_3$  composite with homogeneous distribution of oxide particles was fabricated by magnetic pulsed compaction of passivated aluminum nanopowders preheated up to 500°C. The sintered composite possesses a full density and high microhardness (about 2 GPa), which are stable up to 400°C. The tensile strength of a material exceeds 350 GPa at room temperature and slightly decreases with increasing temperature. The intensive

# **MECHANOCHEMISTRY AND MECHANICAL ALLOYING 2003**

compression of nanopowders during MPC is seen as an effective tool for the formation of nanostructured MMC of other types.

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