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Mechanical activation of metallic powders in planetary ball mills: multi-scale modeling and experimental observation

O Politano[1], A Fourmont[1], S Le Gallet[1], F Baras[1], A A Nepapushev[2], A S Sedegov[2], S G Vadchenko[3] and A S Rogachev[2,3]

[1]UMR 6303 CNRS-Université Bourgogne Franche-Comté, 9 Av. Alain Savary, BP 47870, 21078 Dijon Cedex, France
[2]National University of Science and Technology "MISIS", Leninsky prosp., 4, Moscow, 119049 Russia
[3]Merzhanov Institute of Structural Macrokinetics and Materials Science, Russian Academy of Sciences (ISMAN), Chernogolovka, Moscow Region, 142432 Russia

E-mail: rogachev@ism.ac.ru

Abstract. Ball motion and microstructure formation in the mixtures of Al with metals (Ti, Ni) during high-energy planetary ball milling are studied by means of high-speed video recording, scanning electron microscopy and computer modeling, including molecular dynamics simulation. Possibility of production reactive composite powders with rounded particles for using in selective laser melting techniques has been demonstrated.

1. Introduction

Despite the wide use of High Energy Ball Milling (HEBM) in labs [1], many open questions remain about the efficiency of the process as a function of grinding parameters. It was commonly admitted that the efficiency of HEBM results from high-energy hits with the balls. Recent experimental works contradict this assessment. Indeed, the dynamics of the ball motion during high energy ball milling was investigated in-situ using high speed video recording and the conclusion was that shear deformation due to friction does play a major role in the microstructural transformations [2, 3]. Another important aspect concerns the transformations induced by HEBM in reactive powder mixtures to produce new phase (i.e. mechanical alloying) or to enhance their reactivity. In order to investigate these aspects, we developed experimental technique and multi-scale modeling ranging from the ball motion in the vial to the effect of ball hit on the powder at the nanometric scale. In this work, we investigated the possibility of the production by HEBM of reactive composite powders in the Ti–Al system with various morphology especially with rounded particles with high flowability, which is important for use in selective laser melting (SLM) technology. The reactive composite powders allowed to initiate an exothermic reaction at the relatively low temperature of 650–700°C [4], which can be further decreased by using mechanical activation [5]. After reaction initiation, temperature increased sharply due to the heat release from chemical reaction, and the products, based on titanium aluminides, were formed. These products can be used as structural materials in aerospace engineering. Comparison of experimental observation with modeling demonstrates reasonable agreement.

2. Experimental and modeling

The in-situ images of milling (Activator-2S) were obtained using high-speed video camera MIRO-310, and the milling modes were described as a function of milling parameters (number of balls, size of the balls, vial and disk angular velocities). HEBM of the Ti+Al mixtures was performed in the planetary ball mill «Activator-2S» («Activator», Russia), with variation of the rotation speed of the planetary sundisc (ω_1 = 0–900 rpm) and jars (ω_2 = 0–2800 rpm). The ratio $K = \omega_2/\omega_1$ was varied from K = 1.0 to K = 2.0, which led to different regimes of balls movement in the jars. Initial powder mixture was loaded in the jars along with the milling balls. Volume of the jar was 250 ml, ball to mixture mass ratio 20:1, diameter of the steel milling balls was varied from 2 to 15 mm. HEBM was conducted in the argon atmosphere (4 atm). For the sake of video recording, the balls were placed in specially designed flat jars, without powders. In the modeling we focused on the case of HEBM with a small number of balls (1–5). The trajectory of grinding balls was calculated using discrete element modeling (DEM) in the conditions corresponding to experiments [6]. The behavior of a grinding ball was characterized and classified as stagnant, centrifugal or free-flying. In each case, the associated processes were evaluated. In the case of a free-flying ball, the number of collisions between the balls or between the balls and the walls was estimated. In the case of a stagnant or centrifugal ball, the sliding/rolling ratio was obtained. In the case of two stagnant balls, the specific rolling was investigated. In order to better understand the milling process at the miscroscopic scale, molecular dynamics simulations were carried out using the LAMMPS code [7].

3. Results and discussion

3.1. Experimental results

Figure 1 represents positions of the milling balls during HEBM at different values of K. One ball always locates near the farthest point, as compared to the axis of sun disc, due to centrifugal force. However, using of several balls (*4* in the example in figure 1) results in complex behavior of the balls due to their interaction (cooperative effect). Striking of the balls were observed at K =1.2–1.5, while friction between their surfaces prevailed at K = 1.0 and 2.0.

Initial Ti powder consisted of irregular particles of different sizes; Al particles had spherical shape and smaller size (figure 2). After HEBM, morphology of the powder particles significantly changes (figure 3). Rounded agglomerates appear after 60 min of HEBM, which become smoother after 120 min. Cross-sections of the agglomerates show that these particles are composed of deformed Ti particles in the Al matrix. Heating of the composite powders in Ar and vacuum showed that exothermic reaction was initiated at ~ 650°C resulting in formation of $TiAl_3$ and residual Ti; after initiation, temperature increases up to 1000–1100°C.

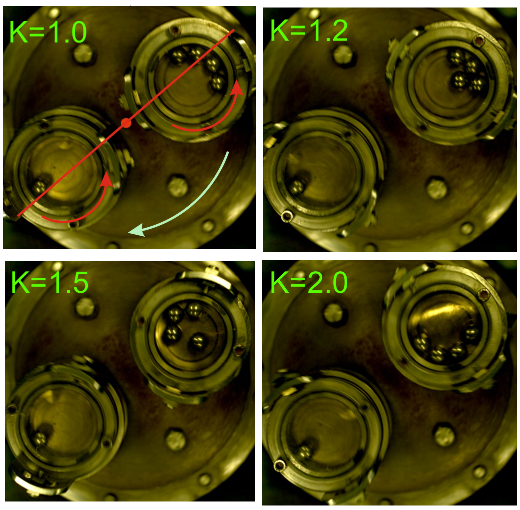

Figure 1. High-speed video frames of the balls motion during HEBM. Balls diameter is 15 mm.

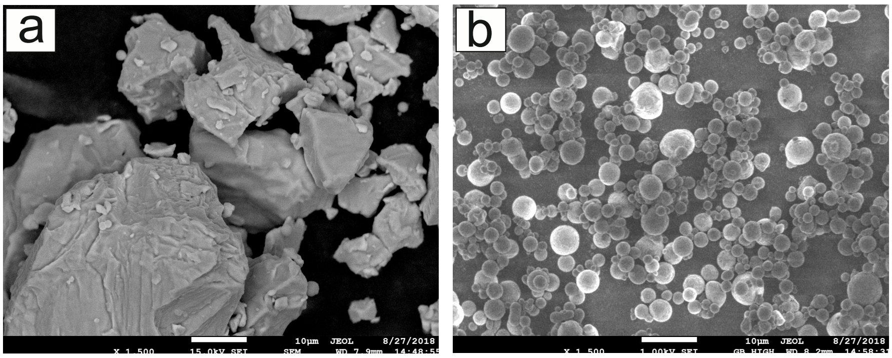

Figure 2. SEM of initial Ti (a) and Al (b) powders.

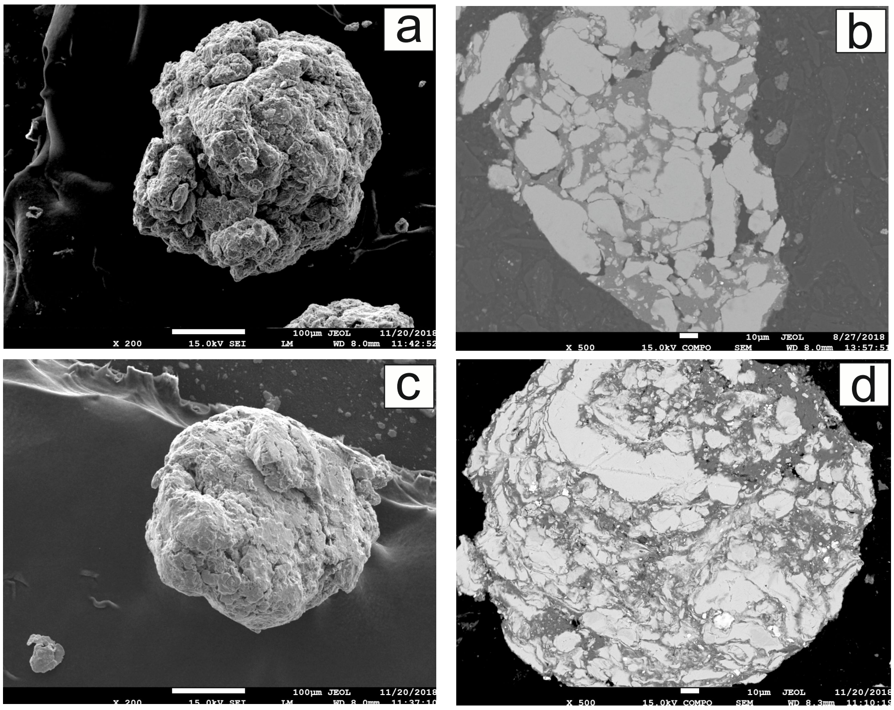

Figure 3. Reactive composite particles Ti/Al formed after HEBM during 60 min (a, b) and 120 min (c, d). SEM, external view (a, c) and cross-section (b, d); light phase Ti, dark grey Al (cross-section).

3.2. Results of modeling
An analysis based on one single ball and two balls (figure 4) demonstrates contradiction with the usual geometrical model [8] and good agreement with experimental observations (figure 1). It proves prevailed role of ball friction in HEBM and provides a guide for more efficient use of HEBM.

The effects due to the ball on a powder particles mixture were analyzed by means of a microscopic approach. The idea here is to develop molecular dynamics simulations in order to study the possible mechanisms (compaction, shear, plastic deformation, welding, fracturing, etc.) that can be observed during HBEM when we vary the ratio *K*. These elementary processes could be incorporated into a Monte-Carlo model to reach long time scale behavior [9]. We considered a set of spherical metal particles submitted to a rapid compression (the combination of Al and Ni was used for this modeling).

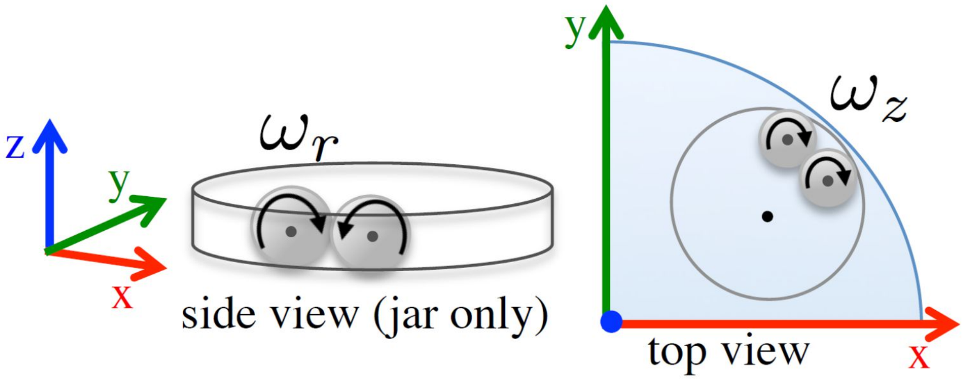

Figure 4. Schematic representation of two balls in the jar. Side view with rotation around the instantaneous radial direction of the ball inside the jar. Top view with rotation of balls around the z-axis. Only a quarter of the disk is represented.

As shown in figure 5, the compression produces a compaction of the powder by removing empty spaces between the particles. Once the particles are compacted, they start to undergo a plastic deformation. Indeed, the ductile Al mostly deforms whereas Ni remains more spherical. An increase of the number of atoms in mixing zones is observed. A detailed atomistic analysis allows us to follow the progressive amorphization, the induced chemical mixing and the possibility of recrystallization due to this first hit. Microstructure of the composite formed due to compression (figure 5) looks similar to the experimentally observed (figures 3b, 3d), despite the difference in scales and compositions.

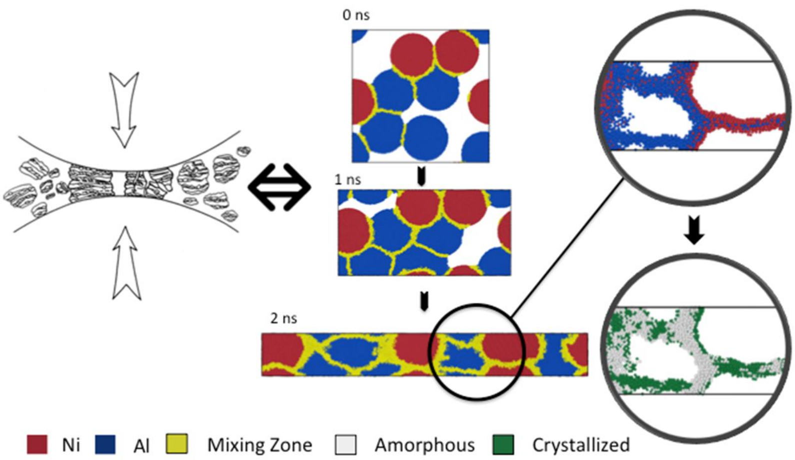

Figure 5. Schematic representation of the impact between two spherical grinding balls and powder (left). Snapshots of the simulated system (right). Initial system with randomly distributed particles in a cubic simulation box (0 ns). Final system after 75% of compression (2 ns). Red particles are Ni and blue ones are Al. Particle diameter is 9 nm. Yellow atoms are atoms that belong to a mixing zone. An atom is considered as "mixed" if one of its nearest neighbors was initially in another particle. For interpretation of thereferences to color in this figure legend, the reader is referred to the web version of this article.

4. Conclusions

Combination of experimental and modeling study of HEBM demonstrates the possibility for producing reactive composite powders (Ti/Al, Ni/Al, etc.) with rounded shape of the composite particles that can be recommended for using in SLM techniques.

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