Influence of mechanical activation duration on morphology, microstructure and microhardness of 3Ni – Al-system powder compound

D A Osipov¹, I V Smirnov¹,², K V Grinyaev¹,² and V V Melnikov¹

¹ National Research Tomsk State University, Tomsk, 634050, Russia
² Institute of Strength Physics and Materials Science SB RAS, Tomsk, 634055, Russia

E-mail: osipov_ff_tusu@mail.ru

Abstract. Investigation of influence of mechanical activation duration in high-energy planetary ball mill on morphology, microstructure parameters and microhardness of 3Ni – Al powder system was conducted. Main stages of transformation of microstructure and mechanical properties are revealed.

1. Introduction

Currently, mechanical activation (MA) is widely used in powder technologies both for milling and for mixing the components of metal powder mixtures, and for the direct synthesis of intermetallic compounds [1 – 4]. Investigation of the influence of MA conditions and duration on morphology, microstructure and strength properties level of precursors and resulting materials is still a topical task.

In the present work, using the example of 3Ni-Al powders mixture, we studied the influence of the duration of MA in energy-stressed planetary ball mills on morphology, microstructure parameters, and microhardness of the resulting precursors.

2. Experimental materials and procedures

The study was conducted using mixture of 3Ni powders (99.85 %) – Al (98 %) after MA in the AGO-2 planetary ball mill in argon. The volume of each of the two steel drums of the mill is 160 cm³. Balls diameter is 8 mm, balls mass in each drum is 200 g, sample mass is 10 g. The centrifugal acceleration of the balls is 400 m/s² (40 g). Water cooling is used to prevent heating in the MA process. The duration of mechanical activation was 1, 1.5, 2.5, 3.5, 4.5, 5.5 and 10.5 minutes.

Morphology of 3Ni – Al system precursors was studied using a Tescan Vega 3 SBH scanning electron microscope (20 kV). Energy dispersive analysis was performed using FEI Quanta 200 3D (30 kV). The microstructure parameters were determined by X-ray diffraction (XRD) analysis on a Shimadzu XRD 6000 diffractometer. X-ray analysis of the phase composition, sizes of coherent scattering regions (CSR) and microdistortions (Δd/d) was performed using the PCPDFWIN database and the full-profile analysis program POWDER CELL 2.4.

Microhardness (Hμ) was determined by the Vickers method on a Neophot 21 instrument with a load of 0.5 N and exposure time of at least 10 s. After mechanical activation, measurements were performed on individual powders and their conglomerates, for which compacts were prepared based on a mixture of precursors with epoxy glue.
3. Results and discussion
The initial (before mechanical activation) state of the powders used was studied in [5]. It is shown that nickel powders have a lamellar form with a developed dendritic structure and are characterized by sizes from 5 to 15 μm. While aluminum powders have a globular shape with a smoothed surface relief, and their sizes are in the range from 30 to 100 μm.

After mixing in a mortar, 3Ni-Al system consists of small and large particles of nickel and aluminum with the initial morphology (Figure 1 a).

![Figure 1](image)

**Figure 1.** Influence of MA duration on morphology of powder compound 3Ni-Al. a) – mortar mixing; MA: b) – 1 min; c) – 1.5 min; d) – 2.5 min; e) – 3.5 min; f) – 4.5 min; g) – 5.5 min; h) – 10.5 min.

Powder after MA lasting 1 minute is characterized by the combination of fine powder into larger lamellar conglomerates (Figure 1 b), which consist of both the original powder and fragments of their milling. The size of such conglomerates reach a length of 200 μm, and the width is about 50 μm.

With an increase in processing time of up to 1.5 minutes (Figure 1 c), both the processes of milling the powders into smaller powders and their combination into large (about 100 μm) conglomerates of a highly distorted form still occur. Based on the data of scanning electron microscopy, including the results of energy dispersive analysis, it was established that fine nickel powders are hammered into soft aluminum particles and their conglomerates. Thus, already at the initial stages of processing, the processes of mechanical mixing of the initial mixture components are activated.

After 2.5 minutes of MA, an increase in the size of conglomerates is observed, which, when compacted, are transformed into a plate-like form (Figure 1 d). The sizes of such conglomerates are 100-150 μm, and their thickness does not exceed several tens μm.

MA lasting 3.5 minutes is characterized by the formation of large (up to 200 μm) compacted conglomerates, which acquire a globular shape (Figure 1 e). In this case, there is a sharp decrease in the number of individual small particles, ranging in size from several μm to several tens of μm. These features indicate the active consolidation processes.

Subsequent processing (4.5 minutes) is characterized by the beginning of intensive dispersion of large conglomerates into spherical particles with sizes ranging from several tens to 100 μm (Figure 1 f). Increasing the processing time to 5.5 minutes contributes to the further grinding of conglomerates whose dimensions do not exceed 50 μm (Figure 1 g). In addition, the appearance of particles of lamellar form, ranging in size from several μm to several tens of μm, is observed.
After 10.5 minutes of MA, large (up to 150 μm) and small (up to several tens of μm) conglomerates are observed, which consist of particles with a size of about several μm (Figure 1 h).

Figure 2 shows the X-ray diffraction patterns obtained from precursors after MA of different duration.

![X-ray diffraction patterns](image)

**Figure 2.** X-ray diffraction patterns after MA of different duration. a) – mortar mixing; MA: b) – 1 min; c) – 1.5 min; d) – 2.5 min; e) – 3.5 min; f) – 4.5 min; g) – 5.5 min; h) – 10.5 min.

As can be seen (Figure 2), an increase in the activation time of precursors is accompanied by a significant broadening of diffraction peaks, which is associated with changes in the size of coherent scattering regions (CSR) and the level of microdistortion (Δd/d). Analysis of the data obtained (Table 1) shows that with MA time increasing from 1 minute to 10.5 minutes, CSR size of the nickel component decreases from 66 nm to 24 nm, and the level of microdistortion increases from 5.2 to 14.3. At the same time, no changes in CSR and microdistortion were detected in the aluminum component in the entire processing time interval.

| t (min) | 1    | 1.5  | 2.5  | 3.5  | 4.5  | 5.5  | 10.5 |
|---------|------|------|------|------|------|------|------|
| CSR (nm)| Ni   | 66   | 69   | 53   | 67   | 38   | 33   | 24   |
|         | Al   | > 150|      |      |      |      |      |      |
| Δd/d·10⁻³| Ni   | 5.2  | 5.2  | 6.2  | 5.5  | 8.4  | 10.9 | 14.3 |
|         | Al   | > 1 |   |      |      |      |      |      |

**Table 1.** CSR sizes and microdistortions level (Δd/d) of 3Ni–Al powder mixture precursors subject to MA duration (t).
Traces of Ni$_3$Al intermetallic phase are observed in precursors after 5.5 minutes of activation, which indicates the beginning of solid-phase synthesis directly in the MA process.

The changes above in the precursors morphology and structure are accompanied by changes in their microhardness (Table 2). As can be seen from the table, in the time interval from 1 to 5.5 minutes, the average values of microhardness vary almost linearly (from 2 to ~ 7.4 GPa). The highest value of $H_\mu = 8.34$ GPa is reached after MA lasting 10.5 minutes. Comparison of microhardness values after 5.5 and 10.5 minutes of MA shows (Table 2) that a twofold increase in processing time contributes to an increase in $H_\mu$ only by 1 GPa. This character of hardening indicates the achievement of values of microhardness close to their maximum.

**Table 2.** Microhardness of 3Ni-Al precursors subject to MA duration [6].

| t (min) | 1   | 1.5 | 2.5 | 3.5 | 4.5 | 5.5 | 10.5 |
|---------|-----|-----|-----|-----|-----|-----|------|
| $H_\mu$ (GPa) | 1.99 | 2.54 | 3.84 | 5.6 | 6.19 | 7.37 | 8.34 |

4. Summary
An increase in mechanical activation duration is characterized both by a change in the morphology and microstructure parameters. The mixing of aluminum and nickel components is accompanied by the transformation of conglomerates consisting of small particles. After 5.5 minutes MA, the synthesis of Ni$_3$Al intermetallic phase is activated in the powder mixture. The transformation of the microstructure and morphology of 3Ni-Al system precursors promotes an increase in the microhardness values with an increase in the duration of activation.

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