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## Determining the effect of process parameters on particle size in mechanical milling using the Taguchi method: Measurement and analysis



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#### ABSTRACT

Micro and nano-particles have been successfully and widely applied in many industrial applications. The mechanical milling process is a popular technique used to produce micro and nano-particles. Therefore, it is very important to improve milling process efficiency and quality by determining the optimal milling parameters. In this study, the effects of the main mechanical milling parameters: milling time, process control agent (PCA), ball to powder ratio (BPR) and milling speed in the planetary ball milling of nanocrystalline Al 2024 powder were optimized by the Taguchi method. Mean particle size  $(d_{50})$  was used to evaluate the effect of process parameters on the mechanical milling process. The orthogonal array experiment is conducted to economically obtain the response measurements. Analysis of variance (ANOVA) and main effect plot are used to determine the significant parameters and set the optimal level for each parameter. The as-received and milled powders were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and a laser particle size analyzer, respectively. The results indicate that the process control agent significantly affects (84% contribution) the mean particle size  $(d_{50})$  while other parameters have a lower effect (16% contribution). The developed model can be used in the mechanical milling processes in order to determine the optimum milling parameters for minimum particle size.

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#### 1. Introduction

Two different terms are commonly used in the literature to denote the processing of powder particles in high-energy ball mills. Mechanical Alloying (MA) describes the process when mixtures of powders (of different metals or alloys/compounds) are milled together. Material transfer is involved in this process to obtain a homogeneous alloy. On the other hand, milling of uniform composition powders, such as pure metals, intermetallics, or prealloyed powders, where material transfer is not required for

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homogenization, has been termed mechanical milling (MM) [1]. The mechanical milling is a complex process which involves the optimization of a number of variables to achieve a desired phase or microstructure. Milling atmosphere, ball to powder ratio, milling speed, milling ball size, milling time, process control agent, starting powder size range, ductility of the initial powders should be considered to achieve the desired products. These parameters influence both the stages of milling and the quality of milled product [2–9]. Fig. 1 shows the evolution of different stages of mechanical milling for a ductile-ductile system [5].

A number of research efforts have been made to improve the experimental conditions of milling process by investigating related issues to assist in choosing suitable mechanical milling conditions. Therefore, Neves et al. [3]



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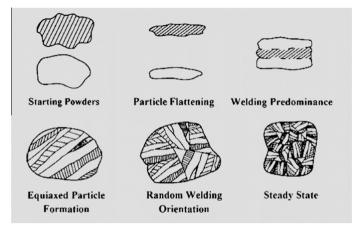


Fig. 1. Evaluation of different stages of mechanical alloying for a ductile-ductile system [5,8].

investigated the effects of milling time and milling speed on the high temperature reaction between Ni and Ti powders. Their experimental results indicate that milling time affects significantly (74% contribution) the enthalpy of the high temperature reaction while the milling speed has a lower effect (25% contribution). Akhgar et al. [10] used mechanical activation to produce the TiO<sub>2</sub> nano-powder. In this study, the parameters such as ilmenite to acid mass ratio, ilmenite to iron powder mass ratio, milling time and initial leaching temperature were selected for optimization of experimental conditions. They reported that the milling time was the most effective parameter on synthetic rutile preparation compared to the rest of the selected parameters. Park et al. [11] used chemical reduction method to synthesis the sodium tungsten oxides (NaWO<sub>3</sub>) particles. In this work, various factors affecting the particle size and standard deviation were analyzed and optimized. They observed that the concentration of sodium tungstate (Na<sub>2-</sub> WO<sub>4</sub>) was the main parameter having significant effects on particle size and the particle size distribution of NaWO<sub>3</sub> particles. Zhang et al. [12] investigated the effects of ball milling parameters on the grain size, median particle size and specific surface area of WC powders. In the Taguchi experiment, they used the weight ratio of ball to powder, size of the milling balls, types of medium, volume of medium and rotation speed as ball milling parameters. They found that except the weight ratio of ball to powder, all other factors are very significant, and the volume of ball milling medium and the rotation speed offer the largest and the second largest contributions in the specific surface area reduction.

In this study, Al 2024 alloy powders were selected to produce nanostructure powders because of its advantages. Al 2024 alloy are extensive used as structural materials in commercial airplanes owing to their good balance of properties including high specific strength, good damage tolerance, formability and corrosion resistance [7]. The objective of this work is to apply the Taguchi method on the optimization of characteristics and to obtain the nanostructured Al 2024 powders by using optimal milling conditions.

#### 2. Experimental details

#### 2.1. Materials and methods

Fig. 2 shows the morphology of the as-received Al powders. The as atomized Al 2024 powders (Gündoğdu Exotherm Company, Turkey) were used as starting materials. The chemical composition of the Al 2024 alloy (in wt.%) was 4.85 Cu, 1.78 Mg, 0.385 Si, 0.374 Fe, 0.312 Mn, 0.138 Zn, 0.042 Cr, 0.005 Ti and Al (balance).

The initial Al 2024 powders were ligamental in shape and these powders were milled by high-energy ball milling. The milling process was carried out in a planetary ball-mill (Fritsch Gmbh, model "Pulverisette 7 Premium line") using a tungsten carbide bowl and balls (10 mm in diameter) at room temperature. Mechanical milling was carried out at the durations of 2, 4, 6 and 8 h, respectively. In order to avoid excessive temperature during milling, the process was stopped every 30 min then resumed when the temperature of the bowl had decreased to room temperature. Bowl temperature was measured between 35 and 55 °C with an infrared thermometer. The Al alloy powders were handled in a glove box chamber under argon gas to prevent atmospheric contamination. To prevent excessive

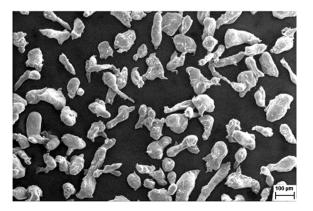


Fig. 2. Morphology of the as-received Al 2024 alloy powder.

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