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# Microstructure and Mechanical Properties of Nanostructured Aluminum Consolidated by SPS

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**Abstract**

*Nanostructured aluminum powders were obtained by means of planetary ball milling with methanol as the Process Control Agent (PCA). The behavior, during milling, was considered measuring the microhardness and grain size at different milling times. Bulk near-full density samples were sintered using the Spark Plasma Sintering technology with different schedules: temperature of 500°C and 550°C, pressure of 30 MPa and 60 MPa and different modes of applying the pressure were changed in order to understand the behavior during sintering. All the samples retained their nanostructure with an increase of the grain size from about 46 up to 70-90 nm.*

**Keywords : Spark Plasma Sintering, Nanostructured Aluminum, Processing Control Agent, Milling, Light Alloys**

## 1. Introduction

The use of light weight alloys (magnesium and aluminum alloys) in the transportation field is becoming highly recommendable as it is environmentally friendly. Anyway, to have their widespread use, the mechanical properties of these alloys should be comparable with steels and titanium alloys; to overcome this problem the use of nanostructured and/or glassy aluminum and magnesium alloys can be suitable. Meantime their use and production has to be cheap and easy enough to appeal to the industry.

For this reason, powder metallurgy is the most promising technology to reach this difficult task; superior mechanical properties at low cost. Obtaining a good nanostructured or glassy powder is quite cheap and simple by means of atomising or milling. However, consolidating and sintering the nanopowder while retaining its small grain size, still remains a problem.

Spark Plasma Sintering is a kind of hot pressing in which a pulsed electrical current flows through the powder. It is able to sinter to full-density, powders at lower temperature and in a very short time [1].

In this paper the production of a nanostructured aluminum powder and its compaction to near full density bulk sample is presented. A pure aluminum powder was milled using methanol as the PCA (process control agent) [2]. The milled powders have been characterized by means of XRD and microhardness. The same analysis have been performed on the sintered samples.

## 2. Experimental and Results

A relatively coarse commercially pure aluminum powder (Sigma Aldrich) was used in the present study. A chemical analysis by means of Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES Spectro Ciros) was performed in order to reveal a purity of 99.84%.

The powder has been milled by means of a Fritsch Pulverisette 5 mill with vials made of tempered steel and capacity of 25 ml; 14 spheres of tempered steel and diameter of 10 mm have been used for each vial; the Ball/Powder ratio was 1:10, so 5.6 g of pure aluminum powder for each vials were used. The acceleration on the spheres was 20 g as described by Fritsch and the milling time ranged from 6 to 168 hours. The vials were always maintained at temperatures lower than 50°C using compressed air.

A quantity of 2% wt of pure methanol was added as the Process Control Agent: the vials were filled with the powder and the PCA without using inert gases or vacuum.

Microhardness was carried out following the E384 ASTM standard by means of an Anton Paar instrument with a load of 5 g. X-ray measurements were conducted using a diffractometer with Cu target for 2θ from 30-140° step 0.05° and the values of the grain size and dislocation density was obtained using a modified Williamson-Hall method [3].

Bulk samples were obtained using a SPS-1050 machine (Sumitomo Coal & Mining Co. Ltd) with pulsed DC voltage (pulse cycle: 12 pulses ON/ 2 pulses OFF, duration 3 ms): the aluminum powder was loaded into the graphite die-punch units to sinter disc-shaped specimens with diameters of 20 mm and thicknesses of 6 mm. To get a better temperature uniformity, a graphite foil was placed between the die and punches. The experiments were conducted in vacuum (2 Pa) and the temperature was controlled using a K-type thermocouple inserted in the die. The inevitable difference in temperature between the die and sample was corrected using preliminary results obtained by putting

inside the sample, a K-type thermocouple and monitoring the die temperature. In this paper the temperature of sintering, Ts, refers to the real temperature at which the sample was produced.

The heating rate, sintering temperature, dwell time and pressure used in the SPS cycles were determined by preliminary tests on the not milled powder [4]. The samples were sintered using many different schedules depending on the sintering temperature (500°C or 550°C), the pressure (30 MPa or 60 MPa) and the possibility of applying the pressure since the beginning of the sintering cycle or at a certain delay in respect to the start.

The milling time could not be as long as was desired. Only the millings up to a time of 48 hrs gave a good yield. Over 48 hrs, the powder particles started to agglomerate and they welded onto the vial walls. This can be due to the decomposition of the methanol and its diffusion in the aluminum to form metastable $Al_4C_3$ carbides. In order to increase the milling time a new addition of methanol was done and a further milling of 24 hrs could be finished: this sample was named 48+24 h.

Figure 1 shows the microhardness for powder samples obtained at different milling times. It can be noted that the nanostructure is immediately formed as after six hours, the microhardness is 125 $HV_{0.005}$ and the grain size obtained, by means of the modified Williamson-Hall method, is 51 nm. When the powder was milled for a longer time, the microhardness further increased; 136 $HV_{0.005}$ after 12 hrs, 146 $HV_{0.005}$ after 24 hrs and 162 $HV_{0.005}$ after 48 hrs.

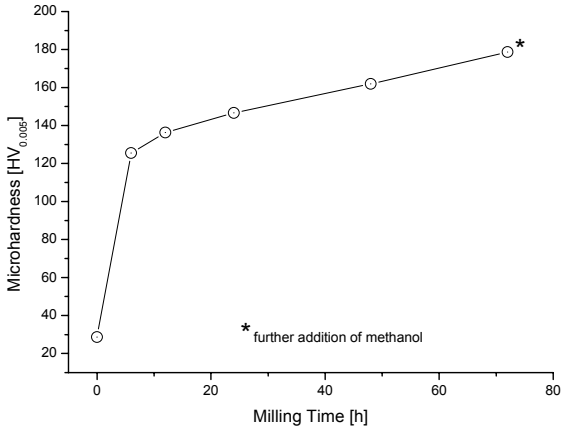


**Fig. 1. Microhardness of the powder obtained at different milling times**

The powder milled for 48+24 h was sintered with the cycles described in the experimental methods and table 1 shows the hardness, the grain size and the density of the different samples.

**Table 1. Hardness, grain size and density for the sintered samples with different schedules**

| *SPS Sample* | *Hardness [$HV_5$]* | *Grain Size [nm]* | *Density [$g/cm^3$]* |
|---|---|---|---|
| 500°C 30 MPa | 80 | 76 | 2.40 |
| 500°C 60 MPa | 99 | 72 | 2.61 |
| 550°C 30 MPa | 113 | 89 | 2.65 |
| 550°C 60 MPa | 127 | 90 | 2.67 |

The hardness is still quite high as a result of the nanostructure which is retained from the results of the XRD analysis. An increase in the grain size was found but it is quite small as a result of the very fast sintering process.

## 3. Summary

Nanostructured aluminum powder was obtained by means of planetary ball milling using pure aluminum and methanol as the PCA. The sample that was milled for 48 hours followed by a further addition of methanol and a milling of 24 hours gave the best results; grain size of 46 nm and a microhardness of 178 $HV_{0.005}$. Bulk nanostructured samples were obtained by means of SPS using different schedules in terms of sintering temperature, pressure and mode of applying the pressure. All the sintered samples retained their nanostructure with a reasonably grain growth. Bulk nearly full-density samples that were obtained at 550°C by means of SPS showed a grain size of 90 nm and a hardness of 127 $HV_5$.

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