



# Microstructural evolution of nanocrystalline chips particles produced via large strain machining during ball milling

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## ARTICLE INFO

### Article history:

Received 30 September 2012

Received in revised form 2 July 2013

Accepted 26 July 2013

Available online 2 August 2013

### Keywords:

Nanocrystalline material

XRD investigations

Microstructure

## ABSTRACT

Nanocrystalline Al 2014 alloy chips were produced from a T6 treated billet by using large strain machining as a severe plastic deformation technique. The chips were crushed and milled for 48 h. The microstructures and morphologies of the chips and their evolution during milling were characterized by means of scanning electron microscope (SEM) and X-ray diffraction (XRD). From XRD investigations the grain sizes, microstrains and dislocation densities of the particles were evaluated. Also, microhardness measurements were performed to estimate the mechanical properties of the particles. It was found that the microhardness of Al 2014 particles increased with increasing the milling time. After milling to 48 h, the particle size reduced to 40  $\mu\text{m}$ , the grain size reduced to 30 nm and the dislocation density increased to  $11.5 \times 10^{15} \text{ m}^{-2}$ . Also, a fine dispersion of the precipitates was formed in the matrix of the as-milled particles.

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## 1. Introduction

Nanocrystalline materials with grain sizes of a few nanometers exhibit unusual properties and have high potential for use in structural and device applications in which enhanced mechanical and physical properties are required [1]. Severe plastic deformation (SPD) is one of the desirable processes used to produce nanocrystalline materials by imposing large amounts of strain into the material. Many researches based on the SPD methods such as equal channel angular pressing have been performed on the precipitation hardenable aluminum alloys [2–7]. The presence of the precipitates in the matrix increases the hardness and the strength of the material and reduces its workability. Therefore, subjecting these alloys to SPD at room temperature is difficult [8]. This is why nearly all of the ECAP researches on the age-hardenable alloys to date have been performed at elevated temperatures or in annealed state.

Plane strain machining is a simple process that could be utilized to deform peak aged aluminum alloys by large values of shear strain even at room temperature [9]. Different amounts of strain ranging from 1 to 10 can be imposed by controlling the machining parameters which are the tool rake angle,  $\alpha$ , the undeformed chip thickness,  $a_0$ , and the deformed chip thickness,  $a_c$  [9–11]. So the shear strain,  $\gamma$ , imposed into the chip can be estimated as [12,13]:

$$\gamma = \cos(\alpha) / (\sin(\varphi - \alpha) \cdot \cos(\varphi - \alpha)) \quad (1)$$

The shear plane angle ( $\varphi$ ) can be obtained as:

$$\tan(\varphi) = ((a_0/a_c)\cos(\alpha)) / (1 - (a_0/a_c)\sin(\alpha)). \quad (2)$$

Another parameter is the cutting velocity which controls the strain rate and temperature rise during process. By controlling machining parameters nanocrystalline materials could be produced in the form of chip. The chip formation offers a suitable framework for studying the effects of large strain deformation in a variety of materials [14]. On the other hand, irregular chips shapes make them inappropriate for use as an engineering material. The present study aims to produce nanocrystalline particle powder from Al 2014 alloy chips by means of ball milling. During machining, nanocrystalline chips were produced from the alloy in T6 temper by selecting an appropriate rake angle. Ball milling was used to reduce the chips sizes and produce fine nanocrystalline particles. The microstructural evolution due to large strain machining and evolution during milling was investigated.

## 2. Experimental materials and procedures

Al 2014 chips with the composition of 3.85% Cu, 0.82% Mg, 0.67% Mn, 0.43% Ni, 0.25% Fe and 0.02% Zn (in wt %) were produced during machining of T6 temper treated rod billets.

The dirty and oxidic surrounding layer of billets was removed and cleaned chips were collected carefully. Machining was carried out at low velocity, less than 20 mm/s, to minimize temperature rise during chip formation. Al alloys have excellent machinability and selecting a suitable rake angle made no harmful side effect on the specimen finishing. Rake angle of  $-5^\circ$  was selected, as it induces a high amount of shear strain, about 5.5. The value of the shear strain was calculated

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by using Eqs. (1) and (2) after measuring the deformed chip thickness ( $a_c$ ) and value of the machining thickness ( $a_0$ ) that were 1 mm and 200  $\mu\text{m}$ , consequently. The details are reported elsewhere [9,12–14]. In order to reduce the chip sizes, the collected chips were crushed by means of multiple pressing instead of in a mortar to exhibit the capacity of the method to be performed in the industries. Fine chips were produced for further ball milling (Fig. 1a). Milling was performed in a planetary machine with a rotating speed of 500 rpm and ball to powder weight ratio of 10:1. Argon gas was filled in the vial and milling was performed under protective atmosphere. To prevent excessive cold welding of the particles, stearic acid was used as process control agent (PCA) and added after 24 h of milling time at a proportion of one weight percent. Sampling was performed every 12 h of milling time and the process was continued for 48 h. Morphologies and sizes of the particles were examined by a Cambridge S-360 type SEM equipped with EDX. In addition, SEM was used to characterize the microstructure of the as-milled particles. A small amount of the particles milled for 48 h was cold pressed accordingly. The cold pressed sample as well as

the sample taken from the as-received sample were polished prior to SEM. Polishing was performed by abrasive papers up to No. 3000 and final polishing was performed using diamond paste. Further microstructural investigations were performed by means of X-ray diffraction using Cu K $\alpha$  radiation. The crystallite size and lattice strain of the chips and of the milled particles were determined by analyzing the X-ray diffraction patterns. The strength of the particles was estimated via microhardness measurements using a Vickers indenter under 100 g load applied for 10 s.

### 3. Results and discussion

In order to investigate the changes in the microstructure and morphology of the particles during ball milling, a small amount was taken after 12, 24, 36 and 48 h of milling time for SEM observations. Fig. 1a and b shows the as-machined chips after crushing and the particles obtained from the chips after 12 h of milling time, respectively. It can be seen that particle sizes less than 4 mm could be obtained from the

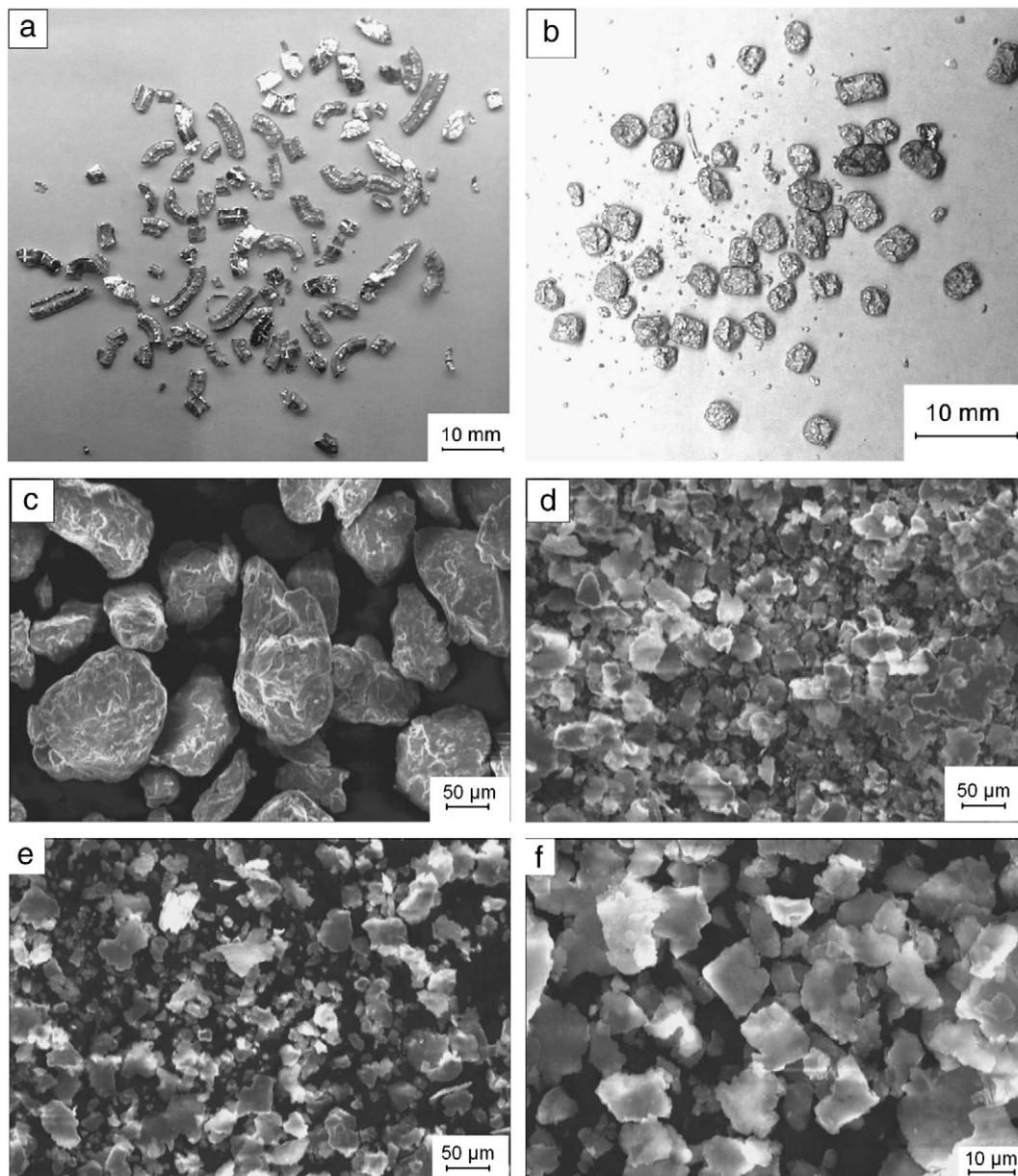


Fig. 1. Changes in the morphology of the particles, (a) broken chips, particles milled for (b) 12, (c) 24, (d) 36 and (e) 48 h. (f) is the higher magnification of 48 h milled particles.

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