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Original Research Paper

Evaluation of structure and morphology of aluminum powder particles milled at different conditions

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ABSTRACT

Micron sized aluminum powder was milled together with different process control agents (PCAs) including ethanol, graphite and stearic acid for various periods of time. The morphology of powder particles was characterized quantitatively using an image processing program and optical microscopy (OM). Work hardening effect on final particles morphology was evaluated by a number of structural characteristics such as dislocation density and crystallite size calculated by the modified Warren–Averbach method. Normal distribution curves for three morphological parameters of Feret diameter, aspect ratio and roughness, were obtained. The results showed that the type of PCA used during the milling operation was much more effective parameter on morphology, dislocation density and crystallite size of powder particles in comparison with the milling time. The specimens with higher work hardening characteristics showed smaller Feret diameter and aspect ratio. Also, ethanol as a liquid PCA found to be more effective comparing with other solid PCAs.

1. Introduction

Mechanical alloying is a powder processing technique, which allows production of homogeneous materials. During milling, the powder particles are repeatedly flattened, cold-welded, fractured and re-welded. After milling for a certain length of time, steady-state equilibrium is attained when a balance is achieved between the rates of welding and fracturing [1]. A PCA is added to the powder mixture during milling to reduce effect of cold welding. The PCA is adsorbed on the surface of powder particles and minimizes cold welding between particles [2]. Addition to PCA, milling time is the most important parameter. Normally the milling time is so chosen as to achieve equilibrium between the fracturing and cold welding rates of the powder particles [2,3].

It has been already shown by Martínez-Sánchez et al. and Suryanarayana et al. that work hardening of powder particles results in their flattening at the first stages of milling [4,5]. By further milling, the flattened particles tend to fracture due to severe work hardening and transform to small particles with nearly sphere shape.

The aim of the present study is to determine the effects of PCA type and milling time on morphology changes of powder particles and their dependence to work hardening, using various quantitative descriptions.

2. Experimental methods and calculations

2.1. Specimens

Commercially pure aluminum powder was milled with different PCAs for 5, 10 and 20 h in argon atmosphere and with a constant ball to powder ratio of 10:1. The milling operation was carried out in a high energy planetary ball mill with hardened chromium steel media and balls. The PCAs added to the Al powder and the times of milling, are shown in Table 1. Samples of the milled powders were cold mounted in epoxy/resin and their section was polished. The OM images were analyzed using the *ImageJ* software (Fig. 1). *ImageJ* is a Java based public domain image processing and analysis program, which is freely available, open source, multi-threaded, and platform independent that can be utilized to develop user coded plug-ins to suit the specific requirements of any conceived application [6]. Using the median (μ) and standard deviation (σ) of each characteristic parameter, the normal distribution was drawn, according to the following equation:

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left(-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right) \quad (1)$$

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Table 1
Samples of Al powders milled with different PCAs.

5 h milling	10 h milling	20 h milling
+0.5 ml Et.[a]	+0.5 ml Et.	+0.5 ml Et.
+0.5 wt% G.[a]	+1.0 wt% S.A.[a]	+0.5 ml Et.+0.5 wt% G.
+1.0 wt% G.	+1.0 wt% S.A.+2.0 wt% G.	+0.5 ml Et.+1.0 wt% G.
+2.0 wt% G.	+2.0 wt% G.	+0.5 ml Et.+2.0 wt% G.

[a] Et. = Ethanol, G. = Graphite, S.A. = Stearic Acid.

2.2. Methods of particle shape characterization

To characterize particles shape, different shape factors, including Feret diameter, aspect ratio and roughness, were gained. Feret diameter is the longest dimension of the particle, which is a good criterion to measure the size of particulates. Aspect ratio is considered as the fraction of major to minor diameters of fitted (Legendre) ellipse. A Legendre ellipse is an ellipse with the center in the object's centroid and with the same geometrical moments up to the second order as the original object area [7]. The Legendre ellipse is depicted schematically in Fig. 2. Roughness may be defined as in the following equation:

$$Roughness = \exp\left(\frac{\sqrt{2}(Perim.)_P}{\pi\sqrt{a^2+b^2}}\right) \quad (2)$$

where a and b are the major and minor diameters of the Legendre ellipse (Fig. 2), respectively.

2.3. Dislocation density and crystallite size calculation

In order to investigate the dependence of work hardening on the final morphology of powder particles, dislocation density and crystallite size were calculated by X-ray diffraction (XRD) results on the basis of line-broadening analysis. The peak broadening in XRD could be caused by the instrument, lattice strain or the crystallite size effects. The instrumental effect could be negligible at low 2θ angles by using a special double crystal diffractometer. Lattice distortion is primarily caused by dislocations and solid solution. In the simplest case where the particles are stress-free, the crystallite size is estimated from a single diffraction peak. But in those cases where stress may be present, a more robust method involving several diffraction peaks is required. In this case, there are a number of conventional XRD peak profile analysis methods such as Williamson–Hall and Warren–Averbach available for use. If the lattice strain is owing to the existence of dislocations (which is common in work hardened materials), the strain would be anisotropic as a result of anisotropy of elastic properties. Recent investigations have shown that the conventional peak profile analyses can be updated by taking into account the contrast effect of dislocations on peak broadening [8,9]. The modified Warren–Averbach procedure has been derived which enables the determination of the grain size (or crystallite size) and the dislocation density [10], as same as the modified Williamson–Hall method. In this study, the modified Warren–Averbach (W–A) method was used to calculate the dislocation density, effective outer cutoff radius of dislocation and crystallite size. According to this method, the logarithmic real part of Fourier coefficient of peak profile could be written as the following equation:

$$\ln A(L) \cong \ln A^s(L) - \rho B L^2 \ln(R_e/L)(K^2\overline{C}) + O(K^4\overline{C}^2) \quad (3)$$

where $A(L)$ is the real part of the Fourier coefficient of peak profile, $A^s(L)$ is the size Fourier coefficient, which for a material with an average crystallite size of D is almost equal to $1\text{-}(L/D)$, R_e is the effective outer cutoff radius of dislocation, $B = \pi b^2/2$ (b is the length of the Burgers vector), $\overline{C}$ is the contrast factor of dislocation, $K = 2\sin\theta/\lambda$, L is the Fourier length and O stands for higher order terms in $K^2\overline{C}$. Plotting $\rho B\ln(Re/L)$ versus $\ln(L)$ and the size Fourier coefficients A^s versus L enable the graphic determination of ρ, R_e and D, respectively [9,10].

Having known the elastic constants of the matter, the average C factor for different kinds of dislocations (pure edge type, pure screw type or mixed type) can be calculated according to Ungár et al. model [12]. By giving into account aluminum's elastic constants as $c_{11} = 108.2$, $c_{12} = 61.3$ and $c_{44} = 28.5$ GPa [11], the average C factors in case of pure edge type were calculated for different reflection orders, which are given in Table 2.

A PANalytical X'Pert PRO diffractometer with Cu Kα1 radiation ($\lambda = 0.15406$ nm) was used for the measurements. The diffraction profiles were measured by a special double crystal diffractometer with negligible instrumental broadening and a resolution of 0.01°.

3. Results and discussion

3.1. Quantitative evaluation of morphology

Typical analyzed OM images are shown in Fig. 3. Normal distribution of morphology characteristic parameters for each sample were obtained, which are shown in Figs. 4–6.

In Fig. 4a, it could be seen that ethanol has a significant effect on particle size reduction, whereas other solid PCAs (stearic acid and graphite) tend to increase the particle size. A same observation was reported elsewhere which milled aluminum powders with PCA of stearic acid tend to increase in size [13–15]. In the case of ethanol a sharp peak is seen while for other two PCAs (stearic acid and graphite), broadened peaks were obtained. It is notable that broadened peak is an indication of wider range of existing sizes. Increase of the milling time in the Al-ethanol sample, does not change the size of particles (Fig. 4b). It was shown that further milling after the steady state equilibrium between fracturing and re-welding of the powder particles, will not change the size of particles [1].

In Fig. 4b, three narrow peaks related to mixture of aluminum with ethanol, all are in same position with the same extent of broadening. The particle size distribution at the stage of steady state for fracturing and cold-welding rates is narrow, because

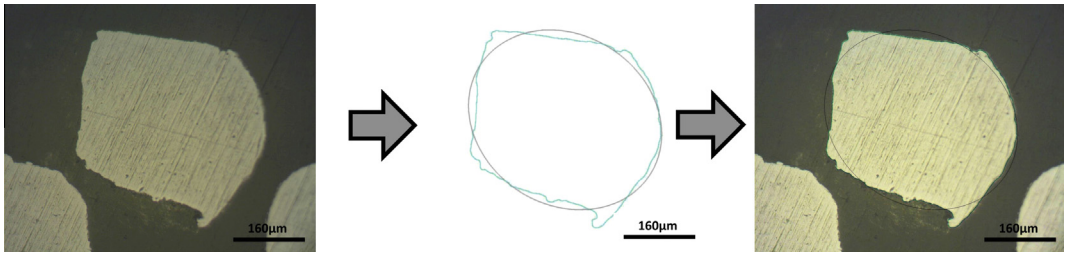

Fig. 1. Progress of morphology analysis by ImageJ.