



Fractal Analysis of the Particles in Cryomilled Nanostructured Alloys

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ABSTRACT

Fracture due to the presence of particles, together with plastic flow instabilities such as necking and shear banding are responsible for the very limited strain to fracture observed in nanocrystalline materials. The sizes, shapes and distribution of these particles always have serious effects on the mechanical properties of nanocrystalline materials. This work is therefore a study of the sizes, shapes and distribution of the particles in three cryomilled nanostructured Aluminum alloys. The result indicated that the predominant particles in the nanocrystalline materials are of regular shapes with $\beta > 0.3$ and $D \geq 1$. It was further observed that the particle distribution maps showed that the particles are clustered with regular backgrounds, thereby creating ease of linkage of the particles.

Keywords: *Fractal Analysis, Particles, Cryomilling, Nanostructures, Aluminum Alloy.*

1. INTRODUCTION

Nanocrystalline metals are of significant research interest because of their high strength. Methods of fabricating nanocrystalline metals include: mechanical alloying, rapid solidification, high pressure torsion, equal channel angular processing, plasma processing, and vapour deposition. These methods can only be used to produce small volumes needed for research purposes but not the large quantities needed for structural applications. This led to the introduction of another form of mechanical alloying called "Cryomilling". In the cryomilling process the milling is done under cryogenic atmosphere i.e an atmosphere of continuous introduction of nitrogen gas. Cryomilling has many advantages over the other methods of production of nanocrystalline metals [1, 2, 3, 4 and 5]. These include:

- (1) Reduction of Oxygen contamination from the atmosphere
- (2) Large convective heat transfer between the particles and the cryogenic atmosphere. Which results in lower particle temperature.
- (3) Reduction in the milling time to achieve nanosize grains

At the nanoscale, the most fundamental properties of materials and machines depend on the size of the nanoparticles or grains. For alloys and composite materials containing regular microstructures a prediction of mechanical properties can be made by a quantitative measurement of features such as grain size, particle size, and spacing etc. This however is not the case where an irregular microstructure is involved because of difficulty in a numerical characterization of the structure. For each microstructure the application fractal geometry offers a

method by which both the individual particle shapes and the mode of distribution of the particles can be fully described in numerical manner [6]. The fractal analysis was used [7] to observe the size, shape, and distribution of pores in Al-V₂O₅ alloyed composite. The analysis revealed that the pores are of irregular shapes, i.e Shrinkage pores, with $\beta < 0.3$. The graph of β against D shows that as β decreases the D values increases. Similarly, the sizes, shapes and distribution of the pores in samples of Al-20%wtMg heat-treated at 470°C for a soaking time of 30 minutes was done [8]. The results show that in the graph of β against D there exist critical values of fractal dimensions (1.0360 and 1.0510) above which any increase in the fractal dimension causes a decrease in the sphericity. Similarly, there exists minimum values of fractal dimension and sphericity ($D = 1.048$ and $\beta = 0.0384$), above which the tensile strength and hardness increases.

Author [9] studied the effect of particle size, morphology and hardness on cold gas dynamics sprayed aluminum alloy castings. From the nanostructure Fig.1, it was observed that the hard cryomilled particles do not experience extensive plastic deformation. Author [10] did a survey of technological developments for fabricating nanostructured metals and alloys. In the survey they examined the nanoparticles in different nanostructures, an example of such nanostructure is shown in Fig.2

Review of published data was done by [11]. Nanostructural Evolution, grain size and other issues were examined for the nanostructure shown in Fig.3.

This research work is therefore a further analysis on the nanoparticles in Fig.1, Fig.2 and Fig.3.

The intention is to use fractal analysis to study the sizes, shapes and distribution of the particles in the nanostructure of the three cryomilled aluminum alloys.

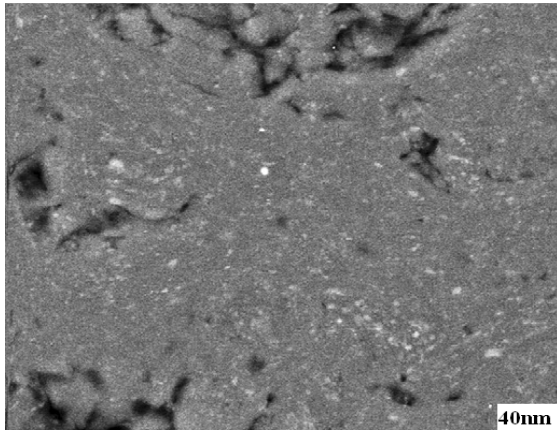


Fig.1: Cryomilled Aluminum alloy1

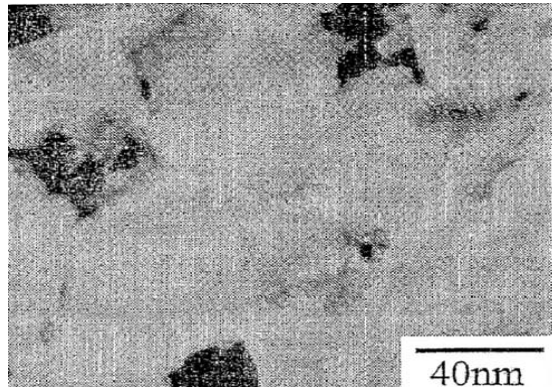


Fig.2: Cryomilled Aluminum Alloy2

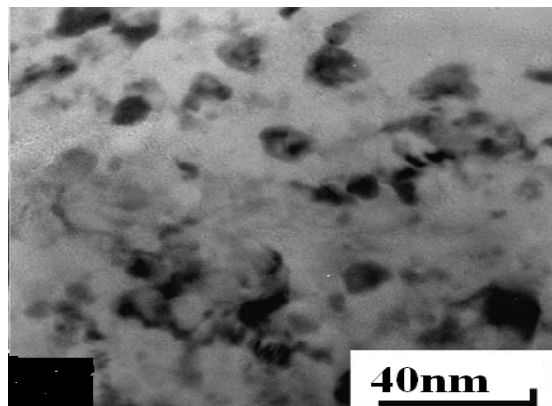


Fig.3: Cryomilled Aluminum Alloy3

2. MATERIALS AND METHODS

Fractal geometry was developed [12] over two decades ago. Its principle is universal in any measurement and has been previously used to numerically describe complex microstructures including graphite flakes and nodules [6, 13

and 14]. The mathematical basis for measuring chaotic objects with the power law modified shall be adopted in this research.

The basic equation is as follows:

$$P = P_E \delta^{D-1} \cdot (1 < D < 2 \cdot \text{and} \cdot \delta_m < \delta < \delta_M) \tag{1}$$

Where P_E is the measured perimeter, P is the true perimeter, δ is the yardstick, δ_m and δ_M are the upper and lower limits respectively, for any shape and D is defined as the fractal dimension ($1 < D < 2$). From the above expressions it can be deduced that the true perimeter is actually a function of the yardstick for measurement. The smaller the yardstick used, the more accurate the measurement. This study intends to use the average of four different yardsticks for better accuracy.

The fractal dimension, D , therefore describes, the complexity of the contour of an object (Fig.4). It can be more practically called the roughness [15].

When $\delta < \delta_m$ the measurement is not sensitive to the yardstick chosen, therefore giving a smaller value of the slope, while $\delta > \delta_M$, the size of the yardstick exceeds that of the individual feature being measured so that the measurement loses meaning because the object falls below the resolution limit of the yardstick used for measurement [6].

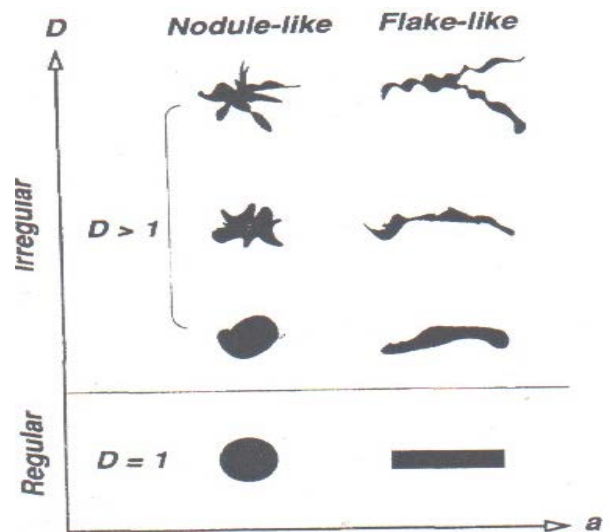


Fig. 4: Illustration of development of irregular shapes based upon Euclidean circle or rectangle. All the shapes have the same area. Source: [6]

Sphericity, β , another dimensionless number, is used together with roughness, D , to describe the shape of the pores formed. It can be expressed as:

$$\beta = 4\pi A_r / P^2 \cdot (0 < \beta < 1 \cdot \text{and} \cdot 1 < D < 2) \tag{2}$$



substituting eq.(1) in (2) gives

$$\beta = (4\pi A_T / P_E) \delta^{2(1-D)} \cdot (0 < \beta < 1 \cdot \text{and} \cdot 1 < D < 2) \quad (3)$$

Where A_T is the total pore area. When $\beta=1$ and $D=1$, a perfect circular shape is formed by the pore in the microstructure. For shrinkage pore $\beta < 0.3$ and for gaseous pores $\beta > 0.3$. As β decreases, the shapes become more elongated showing a departure from a perfect sphere [15]. A study of the distribution of the pores (Fig.4) has been done by different researchers [15 and 16].

The location $1 < D < 2$ represent less regular shapes. It was also discovered that the larger the roughness, the more irregular a pore and thus more stress concentration.

Area of the total pore $A_T = \text{Area of yardstick} \times \text{Number of yardsticks}$

To calculate the perimeter P of the pore, the Slit Island Method (SIM) [17] introduced by [12] is used. It is expressed as :

$$\log_e P = 0.5D \log_e A_T \quad (4)$$

$$P = e^{0.5D \log_e A_T} \quad (5)$$

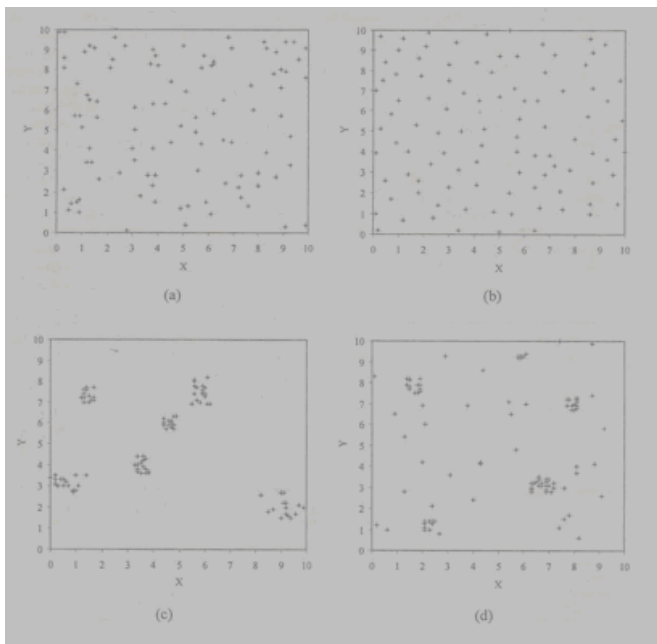
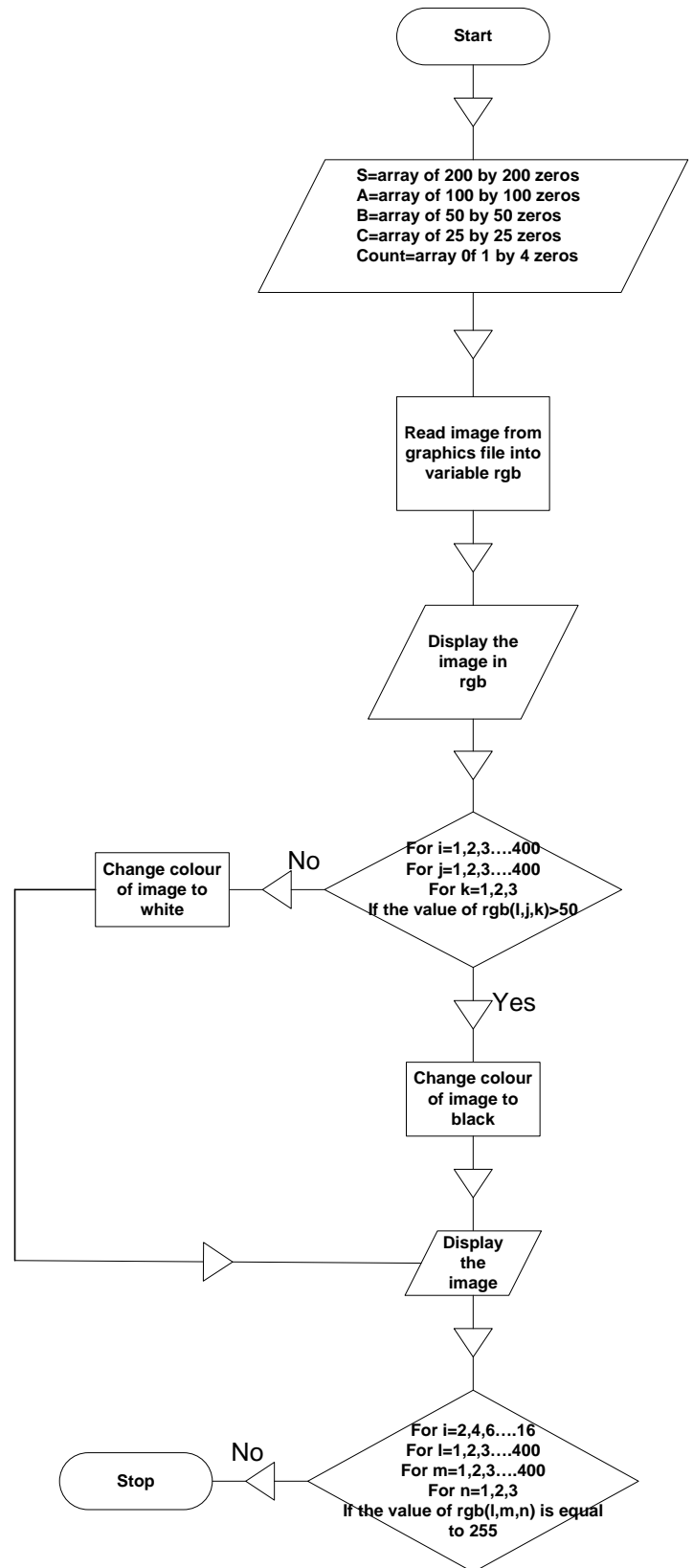
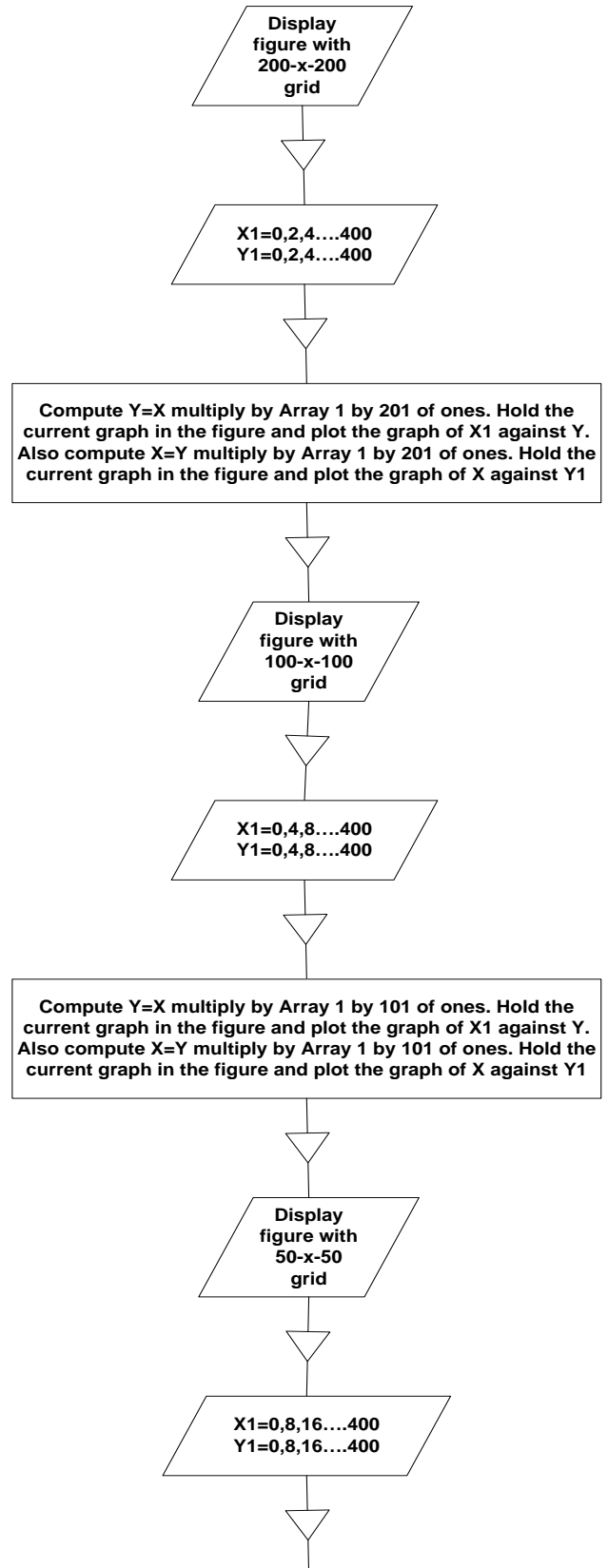
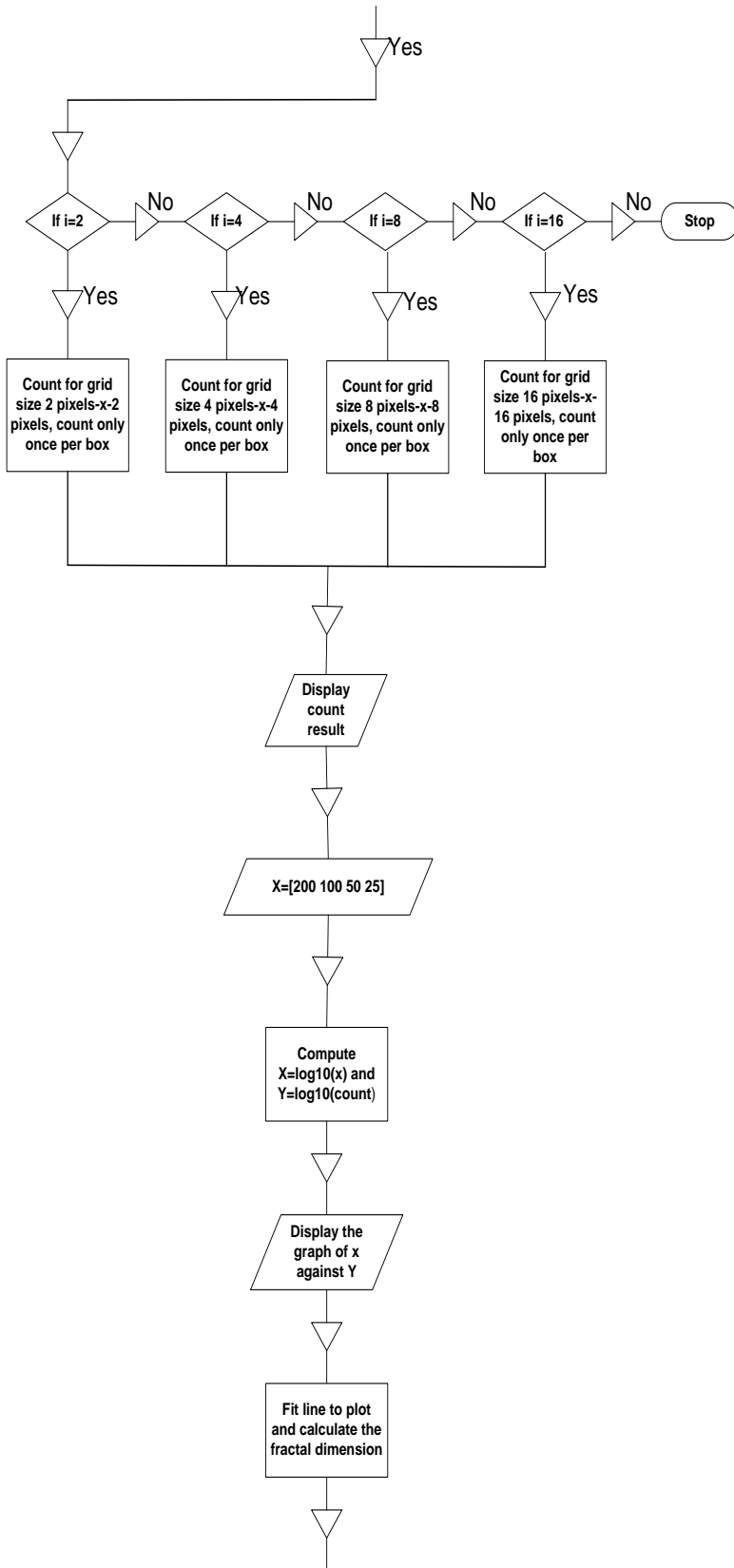


Fig.5: The four common types of spatial point patterns (a) random (b) regular (c) clustered (d) clustered superimposed on random background. Source: [16]

Using the Eq. (1), (2), and (5) above, an interactive software in Matlab programming language is developed to obtain the numerical values of the fractal dimension D and the sphericity β for the microstructures Fig.1, Fig.2, and Fig.3. The flow chart of the Matlab program is Shown in Fig.6.





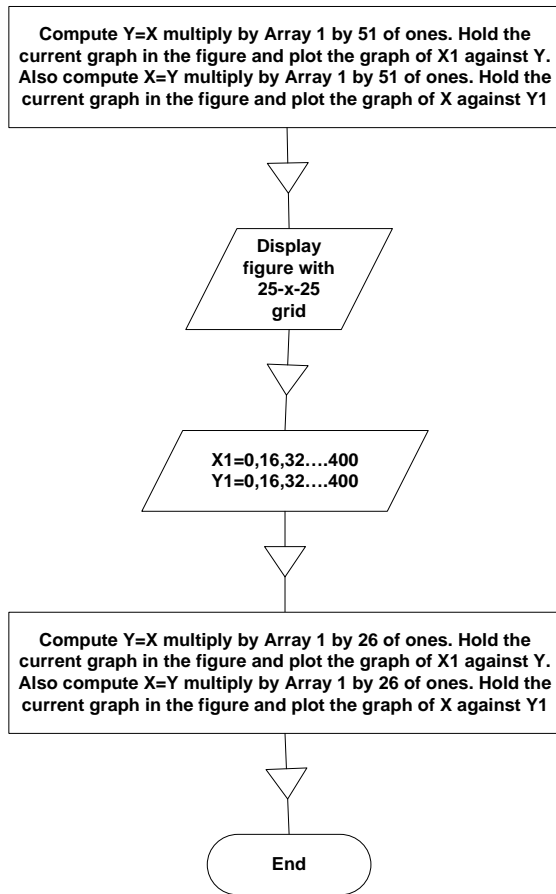


Fig.6: Flow Chart of the Matlab Program

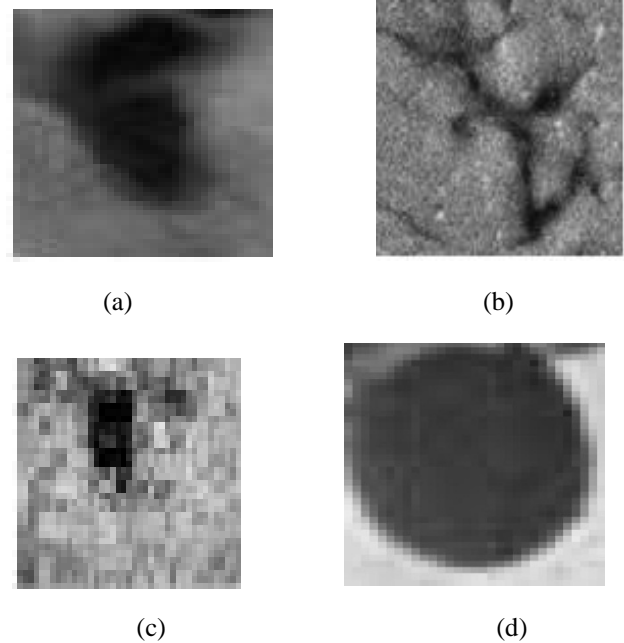


Fig.7: Samples of the Particles from the NanoStructures

The “best” of the particle shapes in alloy1 is the particle with $D = 1.0588$ and $\beta = 0.8363$ while the “worst” shape is that with $D = 1.0602$ and $\beta = 0.2321$. In alloy2 the “best” of the particle shapes is the particle with $D = 1.1030$ and $\beta = 0.9591$ while the “worst” shape is that with $D = 1.5362$ and $\beta = 0.0221$. The “best” particle shape in alloy3 is the particle with $D = 1.000$ and $\beta = 0.9460$ while the “worst” shape is that with $D = 1.3546$ and $\beta = 0.1095$.

3. RESULTS AND DISCUSSION

Figure 7 shows samples of the particles from the nanostructures considered. It was observed that most of the particles are regular in shape with only few having irregular shapes. The degree of regularity or irregularity is determined by the values of sphericity and fractal dimensions shown in Tables 1, 2, 3 below.

The elongated particles were compared to the lamellar structures that arise during cold rolling of wrought Aluminum to high strains with continued deformation taking place within the individual lamellae, producing sub-grains that are roughly equiaxed. This microstructure is thus consistent with the empirical model, but the appearance of high-angle grain boundaries separating elongated regions is not necessarily anticipated. The implication for the cryomilled alloys, though not stated, is that these nanostructures may represent evidence of insufficient milling time. Elongated particles of similar dimensions were also reported as a transient feature in cryomilled Inconel 625, present in samples milled for four and six hours, but not in powder milled for eight hours.

Table1: Fractal Dimension D and Sphericity β of Cryomilled Aluminum Alloy1

S/N	D	B
1	1.0602	0.2321
2	1.0512	0.2667
3	1.0481	0.4451
4	1.0588	0.8363
5	1.1563	0.9034
6	1.3870	0.5435
7	1.1030	0.9591
8	1.2528	0.9724

Table2: Fractal Dimension D and Sphericity β of Cryomilled Aluminum Alloy2

S/N	D	B
1	1.1231	0.6870
2	1.1590	0.8012
3	1.1129	0.9910
4	1.1444	0.3321



5	1.5362	0.0221
6	1.2821	0.3788
7	1.2110	0.3931
8	1.1030	0.9591

4	1.0727	0.8434
5	1.0358	0.8399
6	1.1136	0.9948
7	1.3546	0.1095
8	1.0000	0.9460

Table 3: Fractal Dimension D and Sphericity β of Cryomilled Aluminum Alloy3

S/N	D	B
1	1.0743	0.9596
2	1.0021	0.6578
3	1.0259	0.2861

Figures 8, 9 and 10 show the particle distribution maps for the three cryomilled aluminum alloys. It can be observed that the particles are clustered with regular backgrounds, thereby creating ease of linkage of the particles.

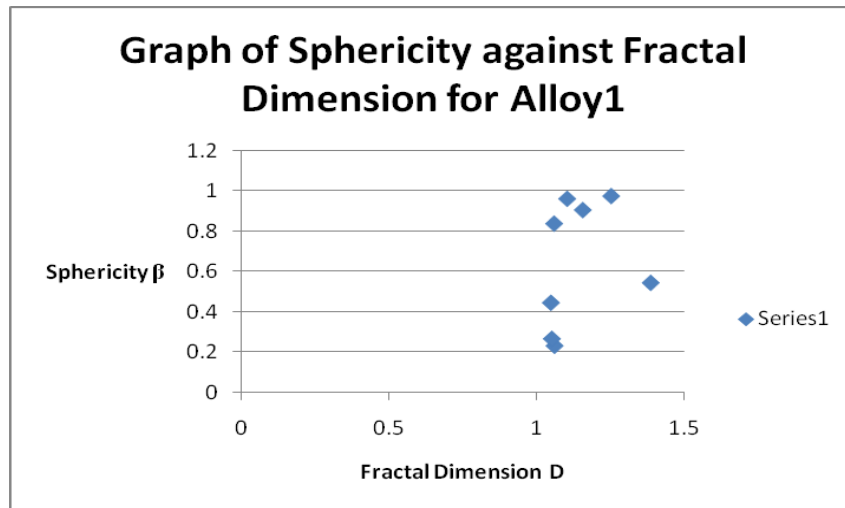


Fig.8: Graph of Sphericity against Fractal Dimension for Alloy1

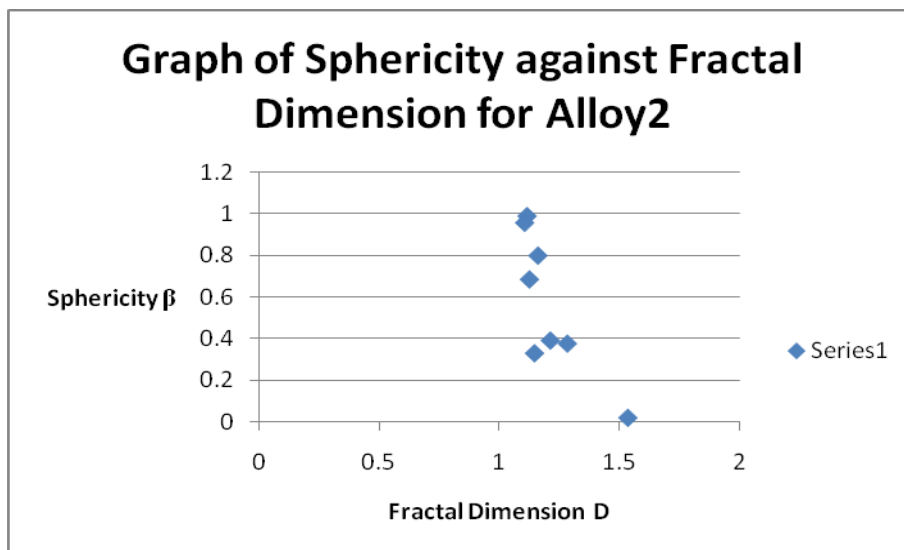


Fig.9: Graph of Sphericity against Fractal Dimension for Alloy2

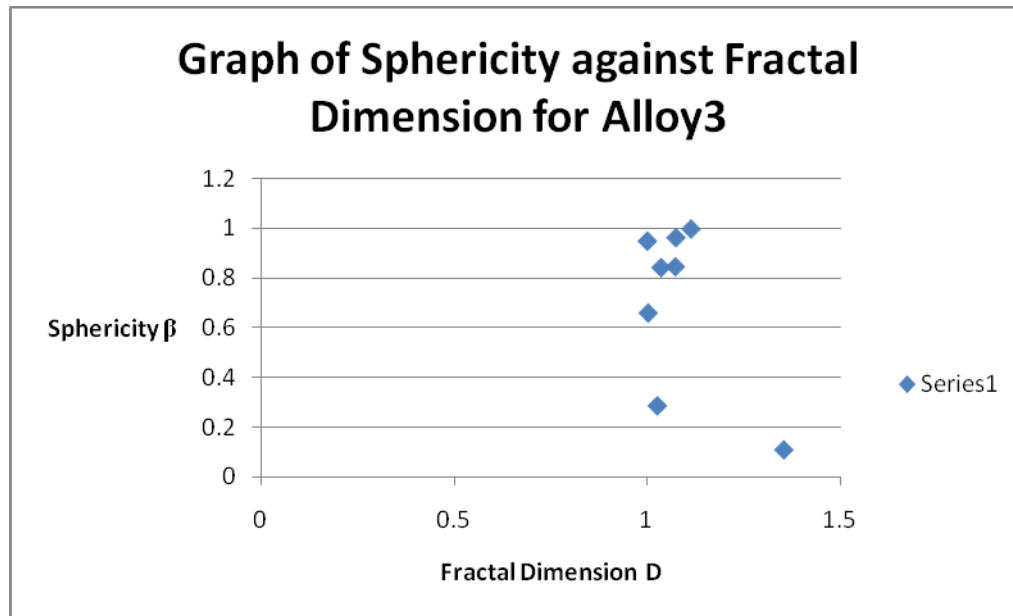


Fig. 10: Graph of Sphericity against Fractal Dimension for Alloy3

4. CONCLUSIONS

The cryomilled powders exhibit grain or particle sizes that are comparable to the minimum particle sizes achieved in mechanical milling. Nanostructured particles obtained in cryomilled powders are mostly of regular shapes compared to those obtained in the microstructure of cast aluminum alloys [7] which are mostly of irregular shapes. Using the fractal analysis the best and the worst of the shapes have also been obtained. Particle distribution maps showed that the particles are clustered with regular backgrounds, thereby creating ease of linkage of the particles.

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