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CONTRIBUTIONS TO COMPUTER-AIDED EVALUATION OF MICROSTRUCTURE FOR PARTICLE REINFORCED COMPOSITES BY MEAN OF IMAGE PROCESSING

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Abstract: *Aluminum matrix composites reinforced with ceramic particles have already proved to be feasible choice when lightweight materials must provide high mechanical properties. Materials samples of composites with reinforcement levels between 0 and 20% in volume have been fabricated using standard powder metallurgy techniques. In order to improve uniformity of particle distribution and remove clustering, additional hot extrusion has been applied at different deformation ratios. Specialized software for image analysis has been used to evaluate correlation between microstructural images and ratios of hot extrusion. The image processing has included image acquisition, image processing, particle detection, particle measurement and classification in relevant granulometric fractions. Resulting histograms have determined strong correlation between particle distribution and deformations ratio and possibility to evaluate effectiveness of plastic deformation upon particle redistribution inside aluminum matrix.*
Keywords: *particle -reinforced aluminum, hot extrusion, particle distribution, computerized microstructure analysis.*

1. INTRODUCTION

Particle reinforcement is a powder metallurgy technology that aims to improve mechanical properties of lightweight alloys, (e.g. aluminum, magnesium or titanium). As a consequence, significant increase of strength, stiffness, wear resistance and fatigue limit may be achieved. Among the fabrication techniques powder metallurgy has a special role, based on some major advantages such as structural homogeneity or possibility to embed even very small particles at reinforcement proportions up to 60%. Since fabrication costs seem to be critical for applications, some general tendencies have been observed [1],[2]:

- Fabrication techniques should be based on standard PM technologies, usually a blending-pressing-sintering route similar to classic materials, that are easy to reproduce and could be implemented with minor technological modifications;
- Both metallic particles and ceramic reinforcements have to be cheap and produced in large quantities, preferably already available on the market;

This last requirement usually produces large difference in particle size between metallic and ceramic powder that could affect material homogeneity, especially when ceramic reinforcements are added in higher proportion. Therefore conventional PM techniques will determine formation of clusters and pores, where ceramic particles are agglomerated inside metallic matrix, as seen in Figure 1. Reinforcement clustering is responsible for dramatic loss of material toughness and ductility, and for this reason secondary processing by mean of high-ratio plastic deformation; becomes necessary for improvement of particle distribution.

So far both clustering of ceramic particles during PM processing and redistribution of reinforcement have been evaluated mostly qualitative. This paper is proposing a more objective tool that could better evaluate microstructural effects of plastic deformation as secondary processing, based on image processing. The computerized processing of the images assumes that a number of mathematical operations have to be followed and logical decisions have to be made in a precise and organized manner.

2. MATERIALS AND METHODS

Samples of composite materials have been fabricated using classic blending-pressing-sintering PM technique. The powder blends consists of a mix of polyhedral-shaped metallic powders (Al - 4,5% Cu, 0,5% Si, 0,7% Mg) with particle diameters between 75 – 95 µm and polygonal silicon carbide (SiC) particles with nominal diameter size of 8,5 µm. These SiC powders are produced at larger scale as grade F800 abrasive powder for polishing suspensions. Volume proportions of reinforcements are 5, 10, 15 and 20%, [3].

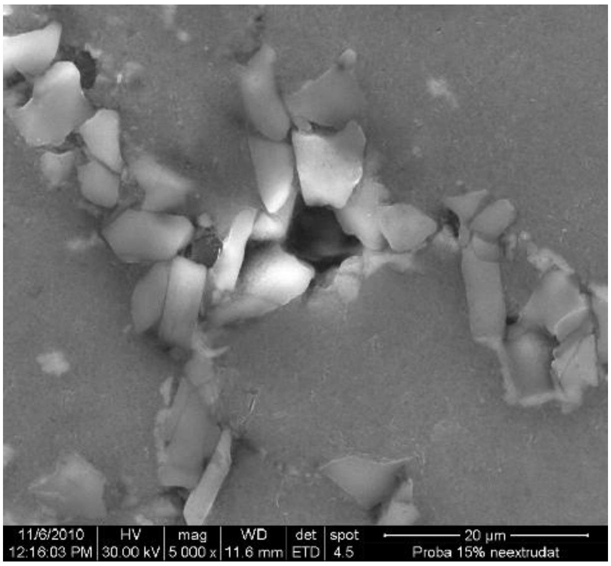

Figure 1: SEM image of clusters of ceramic particles inside metallic matrix, 15% volume of reinforcement proportion, before hot extrusion.

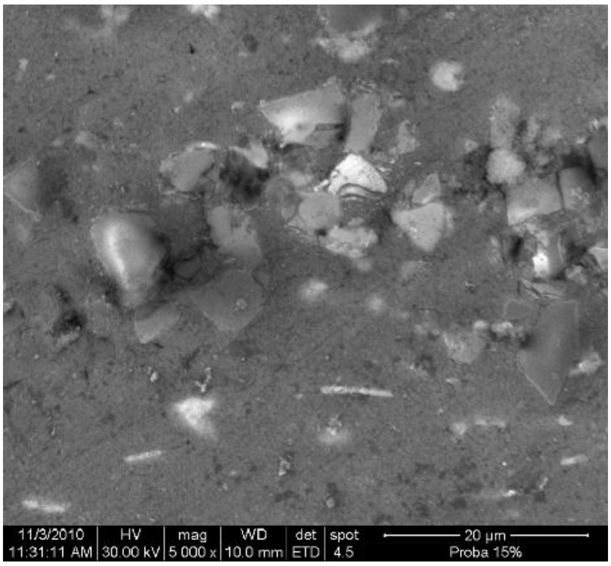

Figure 2: SEM image of clusters of ceramic particles inside metallic matrix, 15% volume of reinforcement proportion, after hot extrusion, i = 2.25.

Redistribution of ceramic reinforcement has been achieved by mean of direct hot extrusion, where samples of 18 mm in diameter have been deformed at diameter values of 12, 9 and 6 mm, which correspond to deformation ratios between incident and resulting section areas ($i = d_o^2/d_1^2 = A_0/A_1$) of 2.25, 4 and 9 respectively.
In order to achieve apparent density around 95% of theoretical density, powder blends have been cold pressed up to 650 MPa. Since ceramic particles prove to have no influence on phase transformations of metallic matrix, a liquid-phase sintering has been applied at 600°C for 30 minutes in argon atmosphere.
Direct hot extrusion has required special attention, since friction between ceramic particles and tool may cause stick-and –slip deformation involving poor surface quality. Therefore a special technique has been developed using a pure aluminum pads, to reduce friction. Optimum deformation temperature has been determined to be 500°C. Remarkably, metallographic investigations determined that pores are no longer present after extrusion, even at low deformation ratio, as seen in Figure 2.
The sequence of technological operations is illustrated in Figure 3, [3] and fabrication details are presented elsewhere [1], [4], [5], [6].

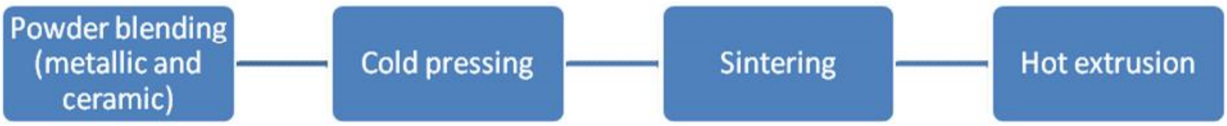

Figure 3: Fabrication route of composite samples.

A proposed variant of the algorithm used to process the images have been presented previously in more detail as in Figure 4, [3].

Figure 4: Algorithm used in image processing, [3].

2.1. Image acquisition.

Image acquisition assumes digitalisation (transformation of analog images into numerical ones) of the virtual image of the real objects obtained using the microscope. During the entire program of test and experiments two microscopes have been used: an optical Olympus BX51M microscope and a SEM FEI Inspect S. In order to digitize the optical images a 4.8 MP discrete sensor array on an Olympus ColorView microscope digital camera has been used. During SEM acquisition both backscattered and secondary electron detectors has been used, on 1 MP images.

2.2. Image Compression.

During all mathematical calculations and transformations only 256 gray levels, monochrome images and 2 levels black and white images has been processed. The optical microscope images are acquired in colours and therefore need to be compressed from 16.7 millions colours (24 bits images) to a 256 gray levels monochrome (8 bits) images. The colour information is stored in 24 bits RGB quantized images using 8 bits for each colour, R, G, B, including 256 levels of gray. The gray tones are obtained when R = G = B. The colour to greyscale image transformation is done by calculating a luminance value, L, for each pixel of the digital RGB colour image using a linear transformation as in Equation 1.

$$L=0.299 \cdot R+0.587 \cdot G+0.114 \cdot B \quad (1)$$

The SEM images has been processed as they were, no compression needed as the depth of the SEM images is 8 bits, only.

2.3. Extraction of Characteristics.

Extraction of characteristic is the most complex step in terms of mathematical calculus. First step in extraction of characteristics is the evaluation of the background illumination. The background illumination is evaluated using morphological operations. Morphology is a broad set of image processing operations based on shapes. Morphological operations are non linear operations that apply a structuring element to an input image in order to generate the output image. The input and output images are the same size and depth. When a morphological operation is applied to a digital image, the value of each pixel of the output image is calculated by means of comparison of the corresponding pixel in the input image with the pixels in its neighbourhood. The shape and size of the neighbourhood defines the structuring element of the morphological operation. The way the shape and size of the structuring element are assigned sets the morphological operation sensitivity to a specific shape in the input image.

2.4. Statistics.

In order to quantify the particles a connectivity test is needed. Connectivity defines which pixels form an object. In a binary image, an object consists in a group of pixels that are set to 1 and are connected to each other. The shape and dimension of an object depends on how connectivity is defined. In two dimensions, standard morphological connectivity is of two types: 4 connected and 8 connected. 4 connected type connectivity assumes that a 1 value pixel is connected to other pixels if at least one of up, down, left or right neighbor pixels are set to 1. The mask for a 4 connected type connectivity test is shown in Ecuation 2. The mask is placed centered on the test pixel.

$$4CTM = \begin{pmatrix} 0 & 1 & 0 \\ 1 & 1 & 1 \\ 0 & 1 & 0 \end{pmatrix} \quad (2)$$

8 connected type connectivity assumes that a 1 value pixel is connected to other pixels if at least one of its neighbor pixels are set to 1. The mask for an 8 connected type connectivity test is shown in Ecuation 3.

$$8CTM = \begin{pmatrix} 1 & 1 & 1 \\ 1 & 1 & 1 \\ 1 & 1 & 1 \end{pmatrix} \quad (3)$$

The masks for connectivity tests can be modified to test for multiple pixel touch as a pixel is set to be a part of an object if a minimum predefined number of neighbor pixels belong to that object. In all processed images a 4 connected type with multiple pixel touch connectivity tests were applied.
All objects have been identified and tagged and the following information was processed:
- area of objects
- equivalent diameter
- position (X,Y) of the mass center.

Deeper statistics will be discussed in the results chapter of this paper.

3. RESULTS

Using commercially available image analysis software (Olympus AnalySIS Five) a complete image processing procedure has been undergone. The image processing has included particle detection, particle measurement and classification and has been done using the following procedure:
- the threshold gray values have been set to the values that enables particle separation from the background information;
- the detection parameters has been defined as follows:
- all particles under 1.5 µm has been considered as noise
- all the border particles has been truncated and counted
- all holes inside particles has been filled
- after the detection of the particles the area of each particle has been measured
- the classification scheme of area has been defined as in Table 1 and the area histograms have been build.
- after the detection of the particles the position of the center of the mass of each particle has been calculated.

Table 1. Area Classification Scheme

Class ID	From [µm^2]	To [µm^2]
1	0	10
2	10	20
3	20	40
4	40	80
5	80	160
6	160	320
7	320	640
8	640	1280
9	1280	2560
10	2560	5120

All acquired images are of 440 x 330 µm overall dimension, in 4:3 picture format. A matrix of 100 boxes has been built, of 44 x 33 µm each. The positions of the center of the mass of each particle have been associated with one of the boxes. For each box two values have been calculated:

$N_{i,j} =$ number of particles in box (i, j), $i = \overline{1,10}$; $j = \overline{1,10}$.

$$A_{i,j} = \sum_k A_k \quad (5)$$

where: $A_{i,j}$ is the total area of particles in box (i, j),
particle $k \subset$ box(i, j),
A_k is the area of particle k.

Two set of three samples have been processed. For the first set of samples the particle to matrix ratio is 10% in volume, but the deformation ratio, i, is different 2.25, 4 and 9. The resulting images are presented in Figures 6, 7 and 9. For the second set of samples the deformation ratio is 9 and the volume of reinforcement proportion is different: 5, 10 and 15%. The resulting images are presented in Figures 8 to 10. As noticed, the 10% volume of reinforcement proportion and i=9 deformation ratio sample belongs to both sets. The average values of number of particles in a box, *N*,the average total area of particles, *A,* standard deviation of *N*, *SD*, relative standard deviation, *RSD*, standard deviation of *A, SDA* and relative standard deviation *RSDA* are presented in Table 2.

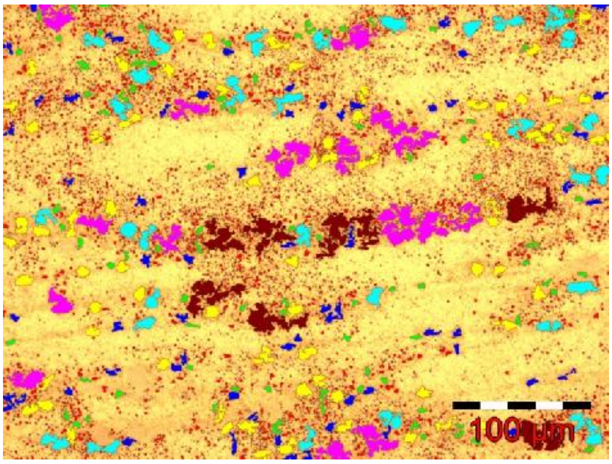

Figure 6: Distribution of area of particles, 10% volume of reinforcement proportion, i=2.25 deformation ratio

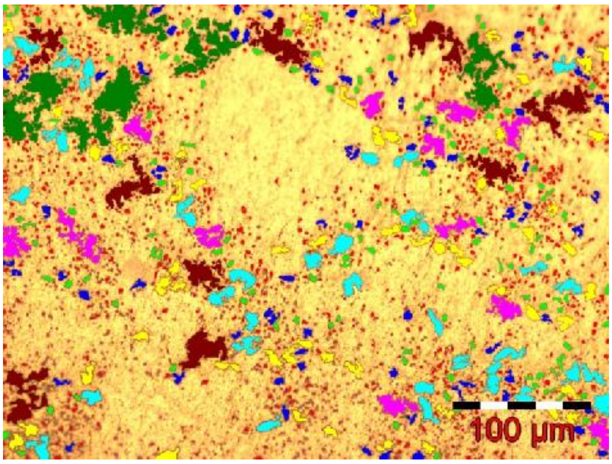

Figure 7: Distribution of area of particles, 10% volume of reinforcement proportion, i=4 deformation ratio

4. CONCLUSIONS

The computer aided method leads to an accurate evaluation of the composite samples in terms of detection of the clusters and the uniformity of the particle distribution within the matrix. In all the samples subjected to optical computer processing clustering of the particles has been observed, but the dimension of the clusters tends to decrease with the increase of the deformation ratio of the sample. The number of clusters tends to increase as the deformation ratio increases. This is a consequence of the fact that the clusters tend to brake in more, but smaller clusters when the composite material is subjected to a higher deformation state. As the deformation ratio increases the particles are distributed more uniformly within the matrix of the samples (standard deviation of $N_{i,j}$ notably decreases with the increase of deformation ratio, table 2).

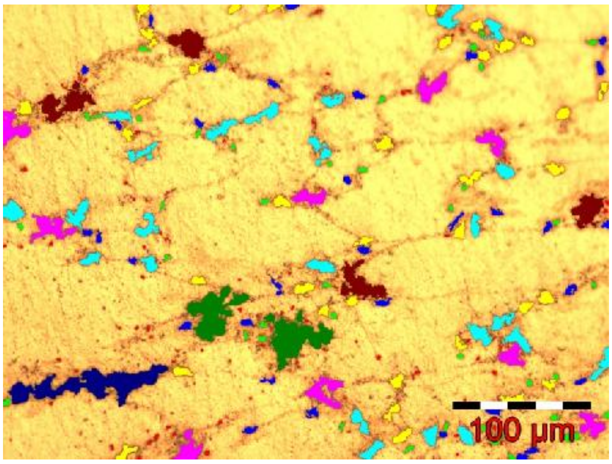

Figure 8: Distribution of area of particles, i=9 deformation ratio and 5% volume of reinforcement proportion

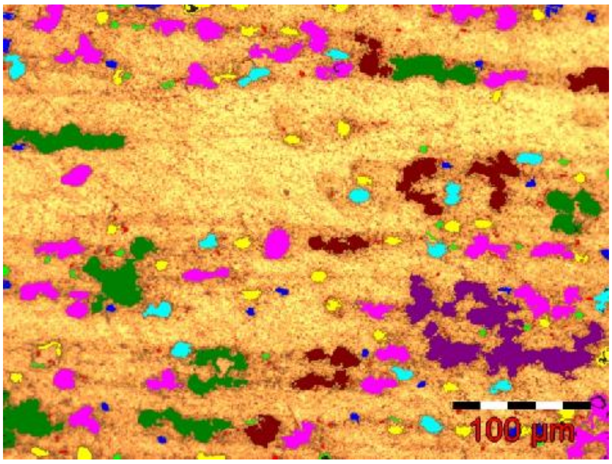

Figure 9: Distribution of area of particles, i=9 deformation ratio and 10% volume of reinforcement proportion

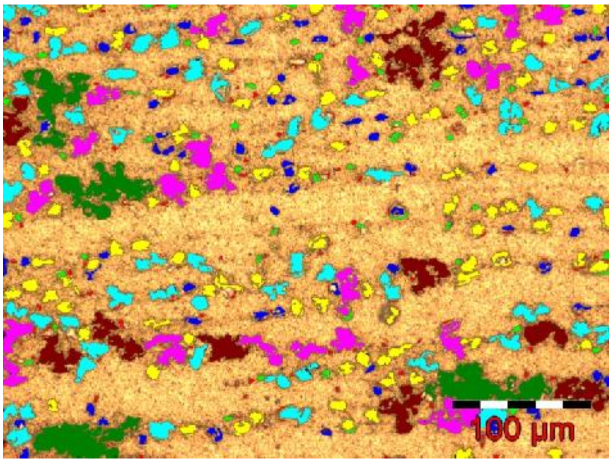

Figure 10: Distribution of area of particles, i=9 deformation ratio and 10% volume of reinforcement proportion

Table 2: Statistics for the analyzed samples

Sample	N [particles]	SD [particles]	RSD [%]	A [µm^2]	SDA [µm^2]	RSDA [%]
10%vol, i=2.25	15.34	8.60	56.06	204.22	157.33	77.04
10% vol, i=4	12.96	7.03	54.24	251.93	249.59	98.67
10% vol, i=9	3.00	2.01	67.00	250.68	421.01	167.94
i=9, 5% vol	2.44	2.19	89.92	133.77	207.72	155.28
i=9, 15% vol	4.61	2.22	48.15	289.22	264.28	91.37

A threshold value of 50% for the relative standard deviation seems to separate the non-uniform distributions from the uniform ones, as a set of 100 boxes are used to evaluate the distribution of the particles.
The tendencies observed on 10 vol.% (the reduction of the number of huge clusters with the increase of deformation factor and the increase in the uniformity of the spatial distribution of the particles with the increase of deformation factor) remain valid for the 5 and 20 vol.%. The phenomenon is more pronounced as the vol.% of the reinforcement particles of the samples increases.

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