AL-1%CU COMPOSITE MATERIAL REINFORCED WITH AL_2O_3 PARTICLES CHARACTERIZATION

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Abstract: An Alumium-1% Cu composite material reinforced with ceramic particles in proportions 5, 10 and 15 % by weight is processed via powder metallugy by mechanical mixture, the materials used are industrial manufacturing residual waste. Once mixed, 300 MPa uniaxial compacting process cilindrical probes were obtained for each proportions, they were sintered at 530º C for 4 hours. Sintered probes microstructure characterization and chemical microanalysis were obtained by Scanning Electronic Microscopy/Optical Microscopy and Energy Dispersive Spectrometry (EDS); furthermore, compressibility porcentage calculations and microhardness tests were carried out. The results show an equiaxial grain microstructure with a high cohesion degree and good reinforcement distribution, in the same way a porcentage of 5-10% indicates a better compressibility porcentage in relation to the microhardness, which could guarantee a good material performance in future conforming process.

Palabras Clave: Aluminium, mechanical mixture, composites, residual waste.

CARACTERIZACIÓN DE UN MATERIAL COMPUESTO AL-1%CU, REFORZADO CON PARTÍCULAS DE AL_2O_3

Resumen: Se procesa vía pulvimetalurgia mediante mezcla mecánica un compuesto Al-1%Cu, reforzado con partículas cerámicas en proporciones 5, 10 y 15% en peso, los materiales utilizados son residuos de procesos de manufactura industrial. Una vez mezclados, se obtuvieron probetas cilíndricas por compactado uniaxial a 300 MPa con cada una de las proporciones, fueron sinterizadas a 530 ºC por 4 horas. La caracterización de la microestructura y los análisis químicos de las probetas sinterizadas fueron obtenidas mediante Microscopía Óptica/Microscopía Electrónica de Barrido y microanálisis químico por Espectroscopía de Energía Dispersiva (EDS); además, se realizaron los cálculos del porcentaje de compresibilidad y ensayos de microdureza. Los resultados revelan una microestructura de granos equiaxiales con un alto grado de cohesión y buena distribución del refuerzo, igualmente para un porcentaje de 5-10% indica un mejor porcentaje de compresibilidad en relación a la microdureza, lo que pudiera garantizar un buen desempeño del material en futuros procesos de conformado.

Keywords: Aluminio, mezcla mecánica, compuestos, residuos.

I. Introduction

Metal matrix materials composite (MMCs) began to be studied in depth in the early 70s as a solution to the demand for better mechanical properties and to achieve weight reduction in aerospace and military systems, group of materials whose properties are custom designed for each application [1].

Many efforts have been directed to the optimization of these materials based on the use of aluminum as matrix, for its attractive low density, excellent resistance to corrosion, wide range of alloys, numerous possibilities of heat treatment and a fairly flexible processing [2-4]. The properties of these composite materials depend on the type of reinforcement, form, quantity, distribution of the phases present in the matrix, among others. In the case of aluminum matrices, oxides, carbides, borides, nitrides or intermetallics particles are incorporated, all of them with high mechanical strength, hardness, elastic modulus and thermal stability [5-6].

Some researchers [7-8], suggest selecting Al2O3 particles to achieve an adequate combination of properties in compounds with aluminum matrix with various intermetallics, as well as modify their content and the size of the reinforcement.

The present study focuses on characterizing a composite material of Al-1% Cu matrix by weight, reinforced with ceramic particles, obtained via powder metallurgy, specifically through mechanical mixing. The aim is to establish the values of density, compaction ratio and microhardness in order to obtain a material that will guarantee sensitive properties for future forming processes. For this purpose, the techniques of Optical Microscopy (MO), Scanning Electron Microscopy (SEM) with chemical microanalysis by Energy Dispersion X-ray Spectroscopy (ESD) were used to reveal the resulting microstructure.

II. Experimental section

To achieve the desired composition of the composite material, it was started with aluminum initial powders, with predominantly elongated morphology and size between 15-150 µm, copper particles of different morphologies: elongated (L≈10-70 µm), angular (L≈ 10-80 µm) and fine globular (D ≈ 1-15 µm), both with purity > 95%. As reinforcement Al2O3 particles with mainly angular morphologies and sizes between 5-75 µm.

The manufacture of the composite material is done with a premixture of Aluminum and Copper powders (Al-1% Cu), in a first stage, followed by the Al2O3 particles, in proportions of 5, 10 and 15% by weight; using 1% by weight of stearic acid (C8H26O2) as a process control agent (PCA), to avoid agglomeration of the particles during the mixing process [9], however, some authors suggest a single step to ensure better metallic properties [10]. The aim was to promote a homogeneous distribution of reinforcing particles within the matrix using a steel container with a suitable seal in a Ratiotrol Boston Gear roller, achieving a cascade effect in the mixture at a speed of 90 rpm for 4 h, without reaching a properly state of mechanical alloy, which could be achieved in high energy mills with higher times and speeds, taking into account the other variables of the milling process and by comparing the results obtained by other authors [11].

Then, cold cylindrical compacts of 22 mm in diameter and 17 mm in height were made, applying a pressure of 300 MPa, using a hydraulic press of max. 50 Tn, looking for these values to produce a compaction through strong deformation and obtaining simultaneously better interfaces contact between the particles more easily incorporating the hard reinforcing particles. These compacts were sintered at 530ºC for 4 h and cooled in the oven until reaching room temperature, using a Nabertherm oven with a maximum capacity of 1280 ºC.

The compacts were characterized microstructurally by Optical Microscopy (OM), using an image analyzer Unitron versamet 3 and a Scanning Electron Microscopy (SEM) with chemical microanalysis by EDS, Esem FEI Quanta 200, the metallographic preparation was carried out with the following steps: roughing (abrasive paper SiC No. 200-600), polishing (alumina 1-0.03µm) and attacked with a 0.5% by volume hydrofluoric acid solution. Likewise for the observation by SEM, the samples were emulsified in ethanol solution, this one is allowed to evaporate and placed in the sample holder with double contact carbon tape. In all cases, working conditions were established using a potential of 20 kV and 15 mm distance from the sample.

Knowing that the density of the samples obtained has a great influence on the final mechanical properties of the developed composite material, it was calculated applying the rule of the mixtures [12], the densities necessary for the calculation of the percentage of compatability of the sintered composite. Likewise, the Vickers microhardness was determined using a HMV SHIMADZU, for the different conditions of the material applying a load of 980 mN and a time of 10 s.

III. Results and discussion

The micrograph of the sintered Al-1% Cu compact matrix, without reinforcing particles obtained by OM is shown in Figure 1a, a microstructure of equiaxed grains with minimal presence of pores is observed, which shows a good homogeneity in the compaction of the material, coinciding with some researchers [13-14] that show that in materials obtained via powder metallurgy exists a relationship between compactness and homogeneity of the mixture.

In Figures 1 (b-d) micrographs correspond to the compacts with Al2O3 particles, in proportions of 5, 10 and 15%, respectively, a microstructure of equiaxed grains is observed which shows a good cohesion between the reinforcing particles and the matrix, this microstructure is mostly observed for a weight ratio of 5% reinforcement. However, as the percentage of reinforcement increases, a greater dispersion of the particles is observed, with a cover microstructure where the definition of the grain boundaries is lost, giving an appearance of agglomeration of the reinforcing particles, this fact is similarly consistent with results obtained in previous investigations [15-16], which coincide in the uniformity of the size of the reinforcing particles to avoid agglomerations product of the pressure and energy applied during the compaction process, which produces a very heterogeneous distribution in the final product and as a result a greater presence of pores.

Equally it can highlight that the appreciable porosity in Figures 1 (b-d) can be influenced by the metallographic preparation because the reinforcing particles in this case of greater hardness than the matrix, as they are not perfectly cohesived, are displaced during the roughing and polishing, which can be evidenced by the geometry of the pores or empty sites.

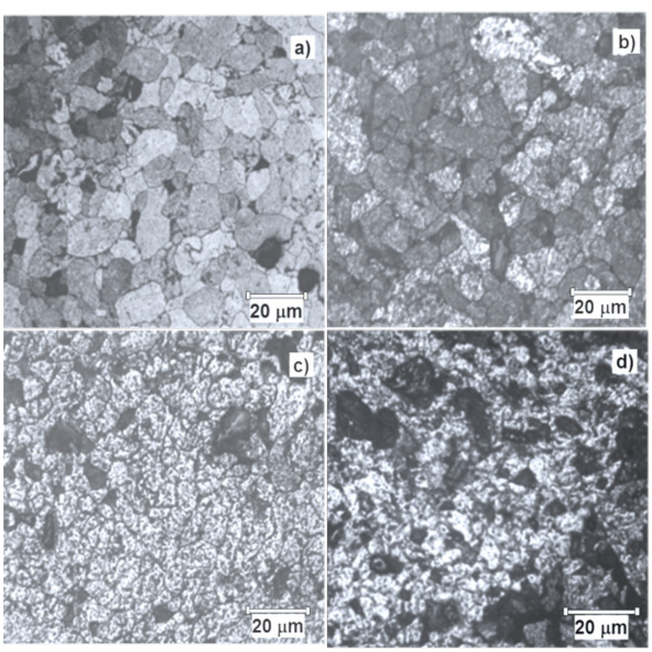

Figure 1. OM micrographs of the sintered compacts: a) Compound Material

Al-1% Cu and b-d with Al2O3 particles 5% (b), 10% (c) and 15% (d).

Attack: 0.5% by volume hydrofluoric acid solution. 100X

In the micrographs obtained by SEM shown in Figure 2a, a preferential location of Cu particles in the grain boundaries of the matrix is observed, this is confirmed by chemical microanalysis by EDS, coinciding with other authors [16-17] that admit the presence of phases clearly defined in the grain boundaries in precipitation studies in Al-Cu alloys; the empty spaces observed in the surface are presumed to be produced during the metallographic preparation, specifically in the application of the chemical attack given the hardness and heterogeneous composition of the oxide particles which was not easy to perform, see Figure 2 (a-d).

Spectrum	O%	Al%	Cu%
3	8,49	39,23	52,28

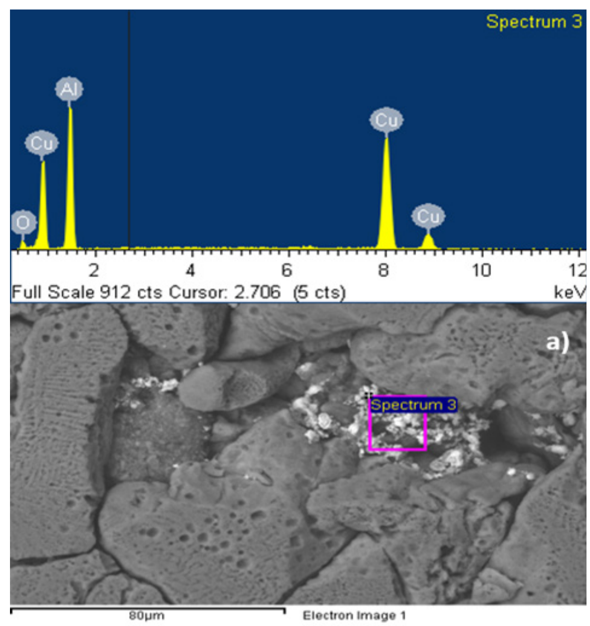

Figure 2a. SEM micrographs with its chemical microanalysis point
by EDS of the sintered compact Al-1% Cu.

Equally we observe that as the percentage of reinforcement increases the particles regrouping perfectly within the microstructure achieving cohesion, located in the grain boundaries, this directly proportional to the aggregate percentage, besides observing, in the corresponding chemical composition, the presence of Elements such as C, Ca, Si, F and Fe that it is inferred are typical of the alumina manufacturing process, without losing of sight of the fact that we are dealing with industrial waste with very specific chemical compositions that could in some way diminish the final properties of the material developed, See Figure 2 (b-d).

Spectrum	O%	Al%	Si%
2	26,63	69,78	3,59

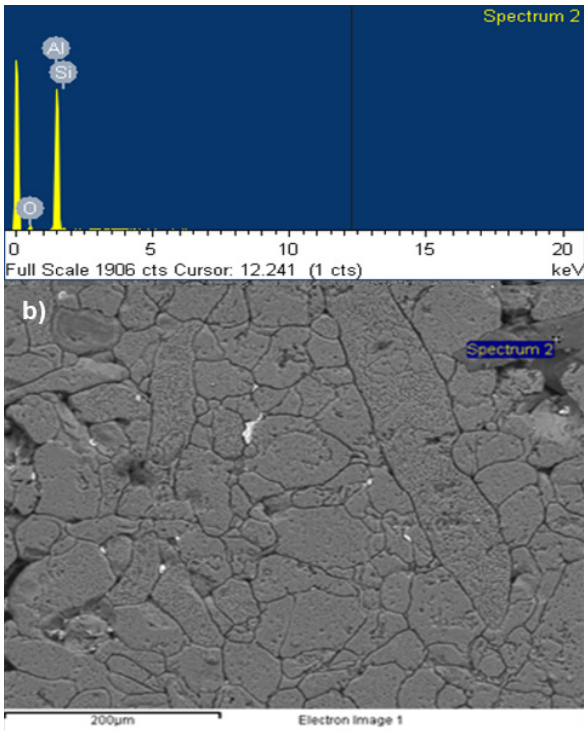

Figure 2b. SEM micrographs with its chemical microanalysis point
by EDS of the sintered compact Al-1% Cu with 5% Al2O3

In the case of study, the needed energy to achieve the atomic mobility of the constituent particles of the powders, to achieve contact or union between them is provided by the temperature of the sintering process that reaches 580 ºC, representing an 80% of the point of fusion of aluminum considered as valid according to previous works [18], who conclude that the ideal sintering temperature is between 70 and 80% of the melting temperature, however, it is below 50% of the melting point of copper and alumina, particles present in the composite material, which could explain the less dissolution of many of these particles located in the grain boundaries, this suggests to do tests with increases in the temperature of the system which could facilitate greater areas of contact and union between the particles.

The undissolved particles shown in Figure 2 (a-d) where we observe perfectly bright particles with high copper content and angular dark particles of alumina, perfectly located in the grain boundaries, even seen in Figure 2 (c-d), that when we increase the reinforcing content are better defined in the grain boundaries, could influence deterioration of the mechanical properties of the material and consequently in the density of the material obtained.

Spectrum	C%	O%	Al%	Ca%
1	-	52,14	47,86	-
2	-	4,95	95,05	-
3	9,82	48,61	41,22	0,35

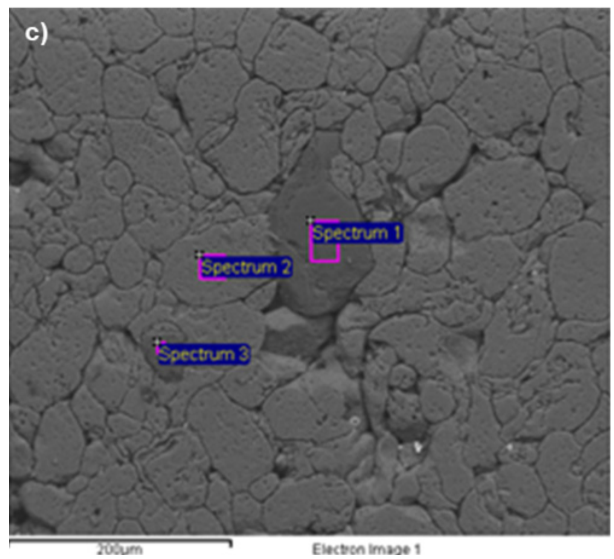

Figure 2c. SEM micrographs with its chemical microanalysis point
by EDS of the sintered compact Al-1% Cu with 10% Al2O3

The alumina particles, undissolved, See Figure 2d, could be embedded in the surface of the aluminum and form agglomerations, which translates into decohesion particles within the material, in this respect some authors [19], determine that the degree of cohesion between the particles is highly dependent on the grinding time of the mixed powders, including their size, in our case it could be inferred that the times of mechanical mixing could be low to achieve a maximum homogeneity in the particles besides influencing their sizes which are quite heterogeneous, that could affect the mechanical properties of the produced compound.

Spect rum	O%	F%	Al%	Fe%	Cu%
1	10,97	6,97	42,86	0,33	38,87
2	5,21	6,97	16,38	-	75,75
3	50,10	-	49,90	-	-
4	-	3,28	96,19	0,53	-

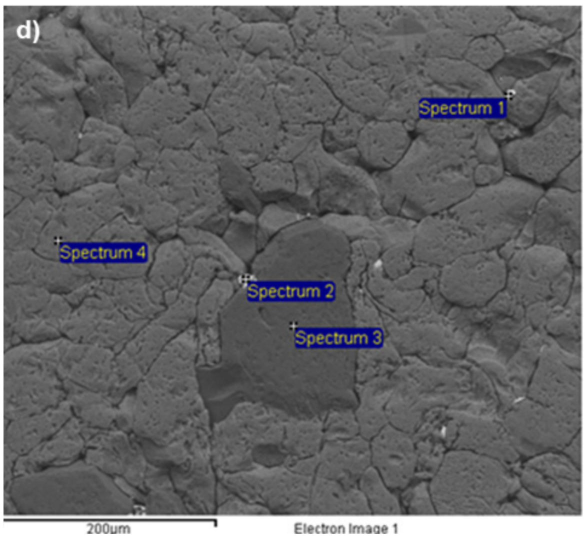

Figure 2d. SEM Micrographs with its chemical microanalysis point by EDS of the sintered compact Al-1% Cu with 15% Al2O3

Table I summarizes the values of relative density and compactability ratio of the Al-1% Cu matrix with the composite material with Al2O3 particles at 5, 10 and 15% by weight, we observe that the value of the relative density for 5% reinforcement increases, however, it tends to decrease as we increase the reinforcement fraction. We also observe that for the greater percentage of reinforcement (15%) the compactability ratio decreases, this behavior can be related to the superficial deformations generated on the aluminum particles after the mixtures and contributed by the morphology and size of the reinforcement, which it is corroborated through the images by OM and SEM, where it is observed that the alumina particles prevent a better cohesion during the compaction and sintering process. This diversity of shapes and sizes of the particles could cause a hardening by deformation of the aluminum that affects the conformability of the compound [19-20].

Despite these characteristics in the particulate material, values of relative densities above 90% and compactability around 60% were reached, suggesting a good cohesion and distribution of the ceramic particles in the Al-1% Cu matrix, under the conditions of processing determined for the manufacture of the test pieces.

Table I
Density and compactability values

% de Reinforce	Density (g/cm^3) a	Theoretica l Density (g/cm^3) b	Relative Density (g/cm^3) c	Compactability Ratio d
Matriz	2,63	2,763	0,952	0,644
5%	2,71	2,822	0,960	0,608
10%	2,73	2,881	0,947	0,632
15%	2,75	2,941	0,935	0,537

a Determined by volumetric calculation.
b Determined by the rule of mixtures (1)
c Density of compacts / theoretical density
d Determined by the ratio ρ compact / ρ aparent x 100

An average Vickers microhardness value of the 24 HV for the matrix was determined, based on eight measurements, which was increased with the reinforcement % added, to values of 27, 31 and 33 HV for 5, 10 and 15%, respectively as illustrated in the graph shown in Figure 3. These values suggest a good compaction of the material despite the characteristics related to size and morphology of the reinforcing particles already described above. These results related to the microstructure and hardness are consistent with other investigations [15-16], which attribute the increase of hardness to the agglomerations of particles during the compaction process, in this case oxide particles whose hardness are much greater than the hardness of the matrix compound.

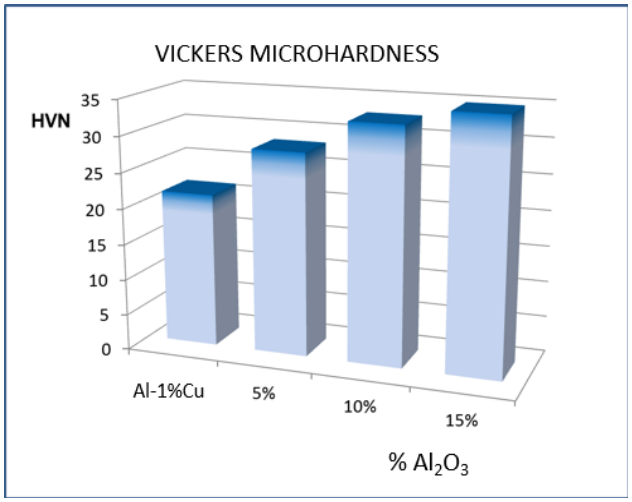

Figure 3. Vickers microhardness values of the sintered compacts of the composite material Al-1% Cu matrix and reinforced with 5%, 10% and 15% Al2O3 particles.

IV. Conclusions

It is feasible to develop by means of powder metallurgy composite materials for manufacturing processes using metallic waste. We obtained compacts in green as resistant enough to be processed and sintered, it was achieving to develop a final composite material with a good relationship between its microstructure and microhardness. The microstructural analysis and the microhardness values obtained from the composite material Al-1% Cu, with the different reinforcement percentage of Al2O3 reveal that for reinforcements of 5% and 10% it was possible to obtain a composite material with a better distribution and consolidation of its microconstituents which could guarantee a better response in future conformation processes. For reinforcing quantities greater than 10%, the percentage of compactability decreased, which did not allow to obtain a consolidated material.

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References

[1]K. Kainer. “Metal Matrix Composites. Custom-Made Materials for Automotive and Aerospace Engineering”. Wiley-VCH:Weinheim Germany, pp. 320-325, Marzo 2006.

[2]M. Taya y R. Arsenault. “Metal matrix composite”. Oxford Pergamon Press, pp. 210-212, Noviembre 1989.

[3]Z. Zhong y N. Hung. “Grinding of alumina/aluminun composites”. Joumal of Matcrials Processing Technology. Vol. 123, pp. 13-17, Abril 2002.

[4]R. Soma, et al. “PM processing of Al-A12O3 composites and thcir characterization”. Powder Mctallurgy. Vol. 46, pp. 219-223, Junio 2003.

[5]J. Ranninger, et al. “Mechanical characterization of AA6061 reinforced with ceramic particles through Mechanical Alloying”. EURO PM Munich, pp. 259-264, Noviembre 2005.

[6]L. Da Costa, et al. “Study of the interaction Matrix Reinforcement through DSC in a AA2014- Ni3Al particulate composite” PM’98: word congress on Powder Metallurgy EPMA Shrewsbury. Vol 1, pp. 645-648, Noviembre 2010.

[7]E. Herranz, et al. “Influence of Nb3Al and NbAl intermetallics additions on properties of hot extruded aluminium powder”. EPMA congress Francia. Vol 4, pp. 51-55, Julio 2002.

[8]F. Velasco et al. “Mechanical properties and wear behaviour of PM aluminium composite reinforced with (Fe3Al) particles”. Pow. Met. Vol 45, pp. 247-250, Mayo 2009.

[9]L. Valencia. “Obtención y caracterización por vía pulvimetalúrgica de la matriz de aluminio reforzada con partículas intermetálicas de CuAl2 y Cu”. Tesis de Grado Doctoral. Universidad del Valle. Santiago de Cali, pp. 125-130, Noviembre 2010.

[10]E. Ruiz, et al. “Influence of blending and particle distribution on Base Aluminium MMC’s.” Word congress on Powder Metallurgy. Vol. 5, pp. 146-151, Febrero 2010.

[11]N. Shoshin, et al. “Preparation and Chtuaetcrization of energetic Al-Mg mechanical alloy powders”. Combustion and Flame. Vol. 128, pp. 259-269, Marzo 2002.

[12]C. Da costa, et al. “Materiales Compuestos de matriz metálica. I parte. Tipos, Propiedades y Aplicaciones”. Revista Metalúrgica Madrid, pp. 179-192, Junio 2000.

[13]V. Salvador y N. Amigó. “Microstructure and Mechanical Behaviour of Al-Si-Mg Alloys reinforced with Ti-Al Intermetallics”. AMPT Leganes Spain. Vol 3, pp. 1499-1508, Septiembre 2001.

[14]F. Velasco, et al. “Mechanical properties and wear behaviour of PM aluminium composite reinforced with (Fe3Al) particles”. Pow. Met. Vol 45, pp. 247-250, Febrero 2002.

[15]L. Cambronero, et al. “Caracterización de la aleación 2014 reforzada con intermetálicos”. Departamento de Ingeniería de Materiales. E.T.S.I. Minas Madrid, pp. 51-55, Septiembre 2014.

[16] A. Cuniberti. "Precipitación en una aleación Al-Cu". Congreso CONAMET . San Nicolás, pp. 345-347, Marzo 2007.
[17]E. Fuentes. "Efecto de la microestructura en las propiedades de materiales compuestos base aluminio". Tesis Doctoral. Universidad Politécnica de Cataluña-España, pp. 146-149. Noviembre 2012.
[18]D. Delforge, Y. Fereira, et al. "Sinterização de uma mistura de cavaco de aço inoxidável com pó do mesmo material. Uma nova tecnologia para a reciclagem de metais". Rev. Esc. Minas vol. 60 N°1, pp. 103-105, Octubre 2007.
[19]P. Yu, C. Kwok, et al. "Enhanced precipitation hardening in an alumina reinforced Al–Cu alloy matrix composite". Rev Scien Direct Composites, pp. 329-331, Octubre 2010.
[20] Z. Wang, M. Song, et al. "Effects of particle size and distribution on the mechanical properties of SiC reinforced Al–Cu alloy composites". Materials Science and Engineering. A 528, pp. 1131–1133, Febrero 2011.