MICROSTRUCTURAL CHARACTERIZATION OF FINE WASTE COLLECTOR FOR USE AS REINFORCEMENT IN COMPOSITE MATERIALS

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Abstract: A characterization is presented of the waste obtained in collector sleeves of electric furnaces, called Fine Waste Collector (FWCs), during the ferroalloy production process in order to evaluate the possibilities of use as reinforcement of metal matrix composite materials. The characterization of the particulate material was carried out through a granulometric classification, in addition with techniques of Optical Microscopy (OM), Scanning Electronic Microscopy (SEM) and chemical microanalysis by EDS, with the purpose of determining size, morphology and elementary chemical composition of the particles, main variables in the evaluation of a particulate material and its use as reinforcement. The results show the presence of mostly spherical morphology particles in a range of size <45 μm, in addition to its chemical composition the presence of important oxides mainly of Mn and Si, typical of the slag of the industrial process is observed. Given the characteristics of the fines, a potential reinforcement for the development of composite materials can be considered.

Palabras Clave: Characterization, waste, reinforcement, oxide, metal matrix.

CARACTERIZACIÓN MICROESTRUCTURAL DE RESIDUOS DE FINO COLECTOR PARA SU USO COMO REFUERZO EN MATERIALES COMPUESTOS

Resumen: Se presenta una caracterización del residuo obtenido en mangas colectoras de hornos eléctricos, denominado Residuo de Fino Colector (RFC), durante el proceso de producción de ferroaleaciones a fin de evaluar las posibilidades de su uso como refuerzo de materiales compuestos de matriz metálica. La caracterización del material particulado se realizó a través de una clasificación granulométrica, además de técnicas de Microscopía Óptica (MO), Electrónica de Barrido (MEB) y microanálisis químico por EDS, con el propósito de determinar tamaño, morfología y composición química elemental de las partículas, variables principales en la evaluación de un material particulado y su uso como refuerzo. Los resultados muestran la presencia de partículas mayoritariamente de morfología esféricas en un rango de tamaño <45μm, además se observa en su composición química la presencia de óxidos importantes principalmente de Mn y Si, típicos de la escoria del proceso industrial. Dadas las características de los finos se puede considerar un potencial refuerzo para el desarrollo de materiales compuestos.

Keywords: Caracterización, residuos, refuerzo, óxido, matriz metálica.

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I.INTRODUCTION

Companies producing and selling ferroalloys use electrometallurgical reduction technology, specifically electric furnaces to obtain a product that combines with other metals to achieve better mechanical properties, using reduced manganese and silicon raw material with metallurgical coke.

During the reduction process, powders are generated that are trapped in a collection cyclone system called fine waste collector, which contains usable minerals, but cannot be reinserted directly into the reduction system, because they would be easily forwarded from electric ovens to the atmosphere, increasing the need for cyclone operation. These dusts that are trapped in the cyclone system are deposited in the company's open pit courtyards, but due to the volume of waste an important environmental passive is being generated in terms of environmental pollution [1], they are also directly proportional to the production process so its generation is continuous and they are being wasted as raw material.

For the explained above, in addition to the new environmental policies implemented in our country, for the use and recovery of materials, industrial waste products, either for reuse in the production process or as possible elements for the development of new materials [2], these fines represent a material of interest due to their size, morphology and composition, suitable for the development of new materials via powder metallurgy [3].

In this powder metallurgical process of obtaining cold composites materials, these fines become a raw material of great interest, specially due to particle sizes, morphological characteristics and mechanical properties, knowing that the tendency is now to use increasingly fine particles, which guarantees better mechanical properties already studied by several authors [4-5] and that are not achieved with conventional casting processes, on the contrary, they are very suitable for this manufacturing, in addition to the low temperatures at which they work in a state solid.

The objective of the characterization of these fines is to know the size of the particles, the morphology and chemical composition that this environmental passive possesses that allows demonstrating their benefits and recommending their use as a possible reinforcement of composite materials of metallic matrix, using as a base techniques granulometric classification, optical and electronic scanning microscopy.

II. DEVELOPMENT

Experimental procedure

The Fine Waste Collector (FWCs) samples were obtained by simple selection since the universe of fines has the same probability of being selected in the sleeves of the company's collection system. Its granulometric characterization was determined using a standard ASTM 100, 200 and 325 mesh sieve battery corresponding to 150, 75 and 45 μm, starting with 300 g of particulate material. The quantitative analysis of the samples focused on the evaluation of the morphology of the dust particles, the traits evaluated were: volumetric fraction, approximate average length and width, applying optical and scanning electron microscopy, the samples were prepared using an epoxy as a binder and resin inlay for better handling, using a UNITRON VERSAMET 3 image analyzer. The analysis by SEM and energy dispersive spectrometry (EDS) was performed with a HITACHI S-800FE microscope, for the preparation of the samples were emulsified in ethanol solution, this was allowed to evaporate and placed in the sample holder with double-contact carbon adhesive tape. In all cases, working conditions were established using a potential of 20 kV and 15 mm distance from the sample.

III.RESULTS AND DISCUSSIONS

The granulometric characterization shows that 87% of the particles have significantly <44μm sizes, represented in Figure 1. This factor can be especially influential in the properties of the material since the particle size is one of the main variables of the particulate material, studied in previous research [5]. The investigations agree that the size can facilitate the welding of the particles in the stages of compactation and sintering powder metallurgy, it can also facilitate the accommodation between the interstices or vacancies of the union of other particles in mixtures for the manufacture of a composite material, also a finer powder represents smaller pore sizes and larger contact surfaces, which guarantees a higher percentage of compressibility indicative of better mechanical properties after sintering.

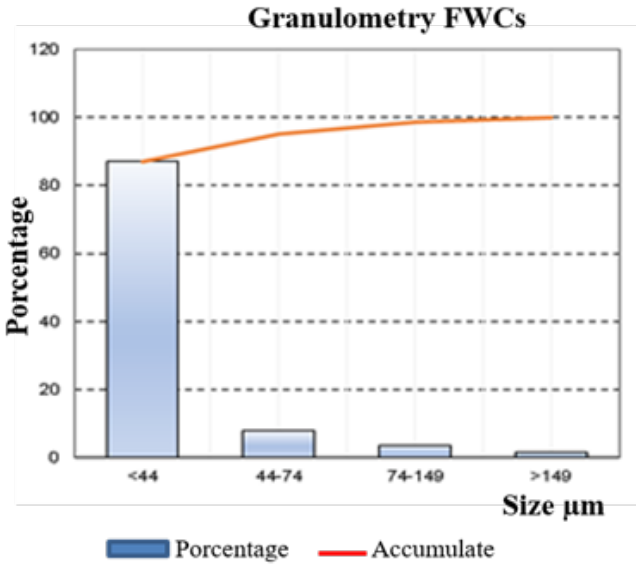


Figure 1. Granulometric distribution for the Fine Waste Collector samples

For the study of the volumetric fractions of the particles present, 300 fields were observed with an area of (1218x778) µm2, determining a volumetric fraction of metal particles in the powder of 2.2%, as can be seen in Figure 2. We observe the high percentage of oxides 97.8%, fully usable as ceramic particles given their size, already represented in Figure 1, which reveal a particulate material with excellent characteristics.

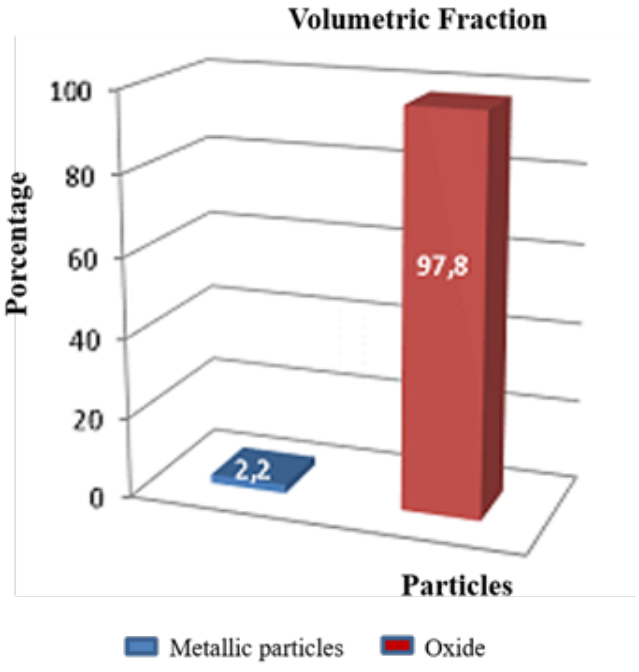


Figure 2. Volumetric fraction of metal particles and oxides present in the powder

In the characterization by OM, represented in Figure 3, metallic particles of ferro-silicomanganese (Mn4Fe-Si3) are observed, named in the company producing ferroalloys, bright of various sizes, in addition, metallic particles possibly opaqued by the adhesion of oxides on their surface , also presence of small pores as a result of the level of adhesion of the particles that make up the dust.

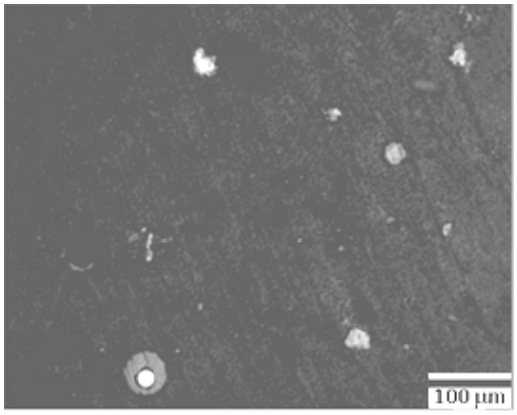
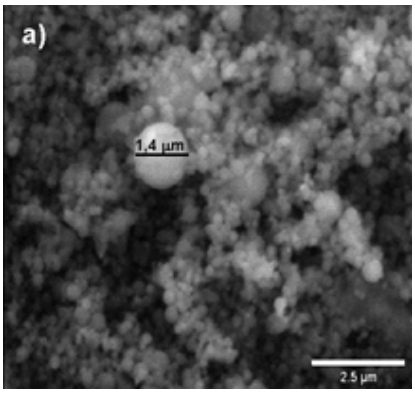


Figure 3. Optical micrographs of the FWCs, the presence of brigh metal particles and pores is observed 200X

Next, the micrographs by SEM, represented in Figure 4, reveal particles of homogeneous spherical morphology, of different diameters in a range of 1.5 to >1 µm. This variability of the diameters of the spheres is attributed to the chemically heterogeneous process that the material undergoes, during the process of calcination in the fusion and subsequent formation of compounds by synthesis for their subsequent crystallization.



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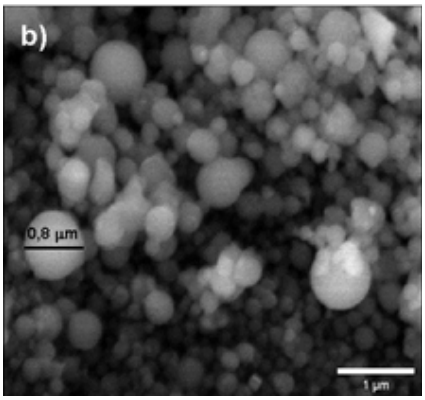

Figure 4. Secondary Electron Micrographs a) Mainly spherical constituent particles. b) Spherical and homogeneously dispersed particles are observed in a matrix of particle agglomerates with smaller sizes.

In the case of the semiquantitative microanalysis obtained, it is observed that the spheres have chemical elements characteristic of organic samples (See Table I and characteristic spectrum in Figure 5), the majority of which are chemical elements C, O, Mn, Si and K, and the minority Zn, Mg, Al, S, Ca, Fe very similarly to conductive organic samples that are considered of interest in previous investigations [6].

Table I. Characteristic elements of FWCs

Element Line	Net Counts	Weight %	Atom %
C K	8.508	49,51	60,99
O K	7.067	36,15	33,44
Mn K	3.518	4,91	1,32
Si K	5.492	3,50	1,84
K K	3.331	2,30	0,87
Zn K	357	0,99	0,22
Mg K	903	0,77	0,47
Al K	938	0,69	0,38
S K	927	0,58	0,27
Ca K	566	0,43	0,16
Fe K	111	0,16	0,04
Ti K	-	0	0
Ba L	0	0	0
Total		100	100

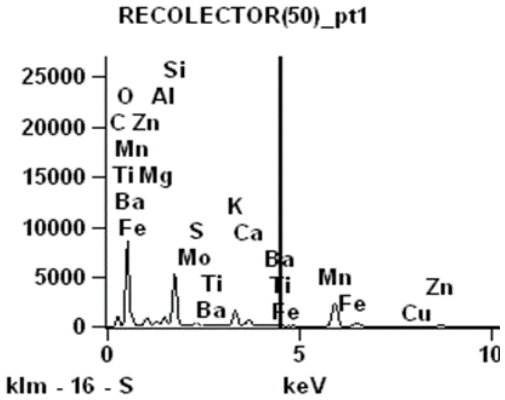

Figure 5. Characteristic spectrum of FWCs

The constituents of the samples were also verified. Performing a spectral analysis by dispersive energy mapping of a FWCs sample, it revealed the main constituents as shown in the images depicted in Figure 6. The primary elementary components are also indicated by intensity of colors as well as the secondary characteristic own of the process of manufacturing.

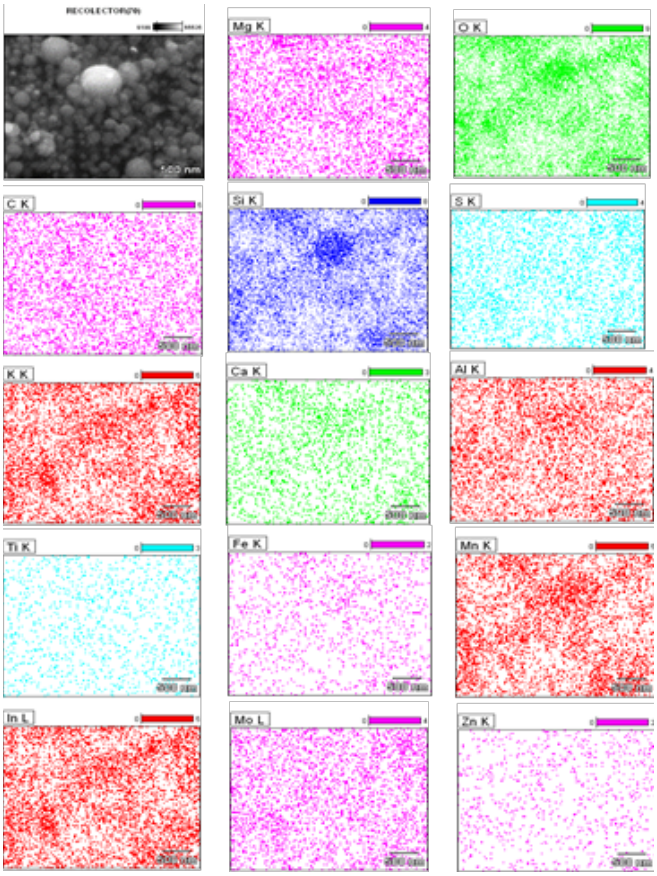

Figure 6. Elemental distribution (mapping) of elements present in FWCs

The images allow corroborating the elemental distribution in the particles, where silicon, manganese and potassium in combination with oxygen, with primary transitions are presented in greater proportions. It is noted with interest that the

majority elements are presented in the form of oxides mainly Mn and Si, which due to their morphological characteristics and size can be evaluated as potential reinforcements in composite materials, referred by other authors in recent research that show these particles as formators of intermetallic that considerably improve the mechanical properties of the materials [7-10].

IV. CONCLUSIONS

1.The Fine Waste Collector has a fairly homogeneous morphology in which ideal spherical particles predominate to guarantee a better performance in powder metallurgy processing.

2.The particle sizes are mostly smaller than 50 µm, which guarantees greater contact surfaces during sintering processes.

3.The elements that predominate in the chemical composition are manganese followed by silicon in the form of oxides, representing among them more than 50% in the composition of the particulate material, which could be perfectly usable as ceramic reinforcements.

4.The characterization techniques used to determine morphological characteristics, size and chemical composition of FWCs, highlight sufficient evidence to suggest their use as a possible reinforcement of material composed mainly of metal matrix.

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