TEM AND SEM STUDIES OF MICRO AND NANOPARTICLES OBTAINED BY ELECTROEROSION DISCHARGE MACHINING PROCESS IN LIQUID N_2.

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ABSTRACT

Nano and micrometric spherical iron particles were produced by the spark-erosion method using liquid nitrogen as dielectric. Transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), standard optical microscopy helped us to find and analyze the spheroidal particles, either hollowed and not with a particular interior and exterior surfaces. No evidences of nitrogen compounds were found.

Keywords: micro particles, nanoparticles, electro-discharge machining.

ESTUDIOS MEDIANTE TEM Y SEM DE MICRO Y NANOPARTICULAS OBTENIDAS POR MAQUINADO POR ELECTROEROSION EN N_2 LÍQUIDO.

RESUMEN

Partículas esféricas nano y microscópicas de hierro fueron producidas por electro-erosión usando nitrógeno líquido como dieléctrico. Microscopía electrónica de transmisión (TEM), microscopía electrónica de barrido (SEM), espectrometría dispersiva en energía (EDS) y metalografía con microscopía óptica común ayudaron a encontrar y analizar partículas esféricas huecas y no huecas con superficies interiores y exteriores particulares. No se encontró evidencia de compuestos de nitrógeno.

Palabras clave: micropartículas, nanopartículas, maquinado por electro-erosión.

INTRODUCTION

In a previous work, [1], we explained the applications of the electro-discharge machining process (EDM) to produce small particles. In few words the main objective of EDM, or spark-erosion developed many years ago, [2], is cutting and holing [3]. The process consists of a swift electric energy discharge in a dielectric, led by a pulsed power source, which strikes on a small area of the material. The surface energy density is very high and the temperature reaches the melting and even the boiling point of the electrodes. A metal vapour bubble is formed on the electrodes‘s surface, acquiring very small vapour droplets which after condensing transform into spherical nanoscopic particles [4]. Part of the remained liquid on the surface is sucked when bubble explodes in the dielectric and suddenly solidifies acquiring a spherical shape. The ejected material as well as the solidified metal vapour form particles that fit a bimodal size distribution [5], corresponding to micro and nanoscopic sizes, respectively. Micro particles are widely used for applications of powder metallurgy and several well-known methods are employed to produce them, [6]. In a previous work, [7] we focused our attention on the study of the ejected material by the EDM process to obtain spherical iron particles using kerosene as dielectric. This method [8] was also applied to produce and study U-Mo spherical particles using pure water as dielectric, [9]. We have observed nanoparticles obtained by electro-eroding Fe in kerosene as dielectric [1]. In order to apply these techniques to nuclear industry, we focused our investigations on the use of dielectrics that avoids the formation of particles chemically reactive with the dielectric as occurred in our previous works, with pure water, [7,9], and kerosene, [1]. The dielectric can be organic, water or liquid gases. In this work we have analyzed by TEM and EDS the particles produced in an current industrial EDM with liquid nitrogen as dielectric with both electrodes manufactured in pure iron.

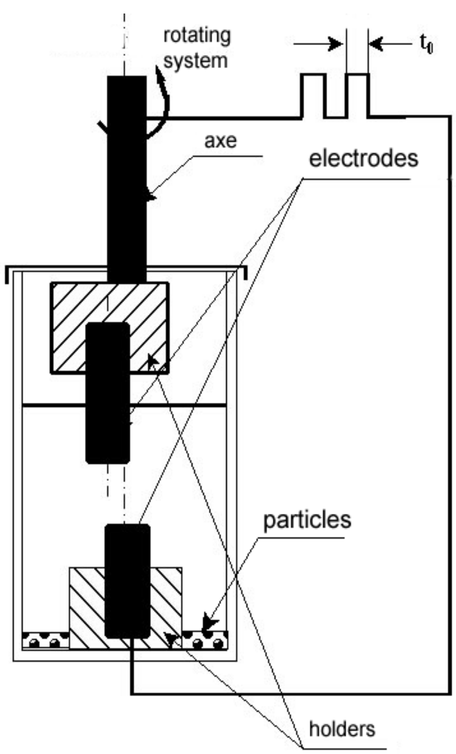

Fig. 1. Device for producing particles by EDM in liquid N_2.

MATERIALS AND METHODS

Our experiments were performed in a commercial EDM machine [10] using rectangular electric current pulses of 9.5 and 25 Amp. The pulse consisted of an active time t_o of 3072 μs and an inactive time t_1 of 340 μs at 80 V, resulting powers of 760 W and 2000 W and energies of 2,33 J and 6.14 J respectively per discharge, so two sets of particles were studied. The dielectric utilized was liquid nitrogen generated in our laboratories. A simple device was specially designed to contain the dielectric and collect the powders resulting from the electrodischarge.

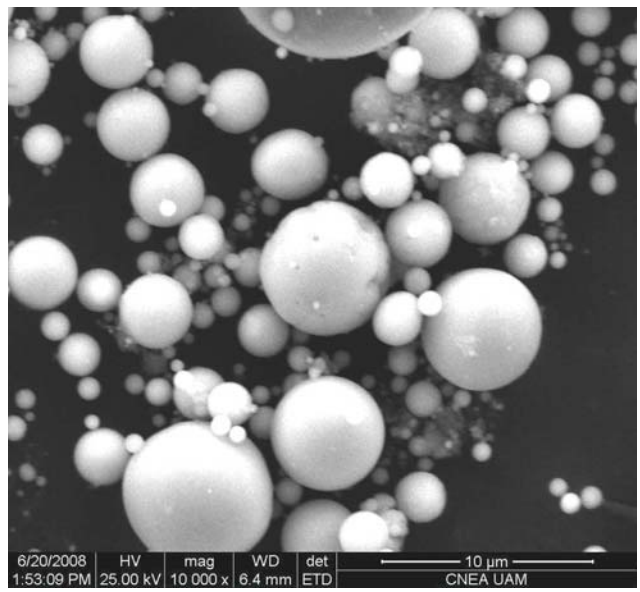

Fig. 2. SEM Micrograph of 9.5 A, 3072 μs particles.

Essentially it consist of a domestic food thermos, see Fig. 1. The studied material in this work arose from the electrodes, called the masterpiece and the tool respectively, being both manufactured in pure iron (Ag <5, Al <5, Ca <8, Cr <2, Cu <5, Mg 2, Mn 1, Ni <5, Si<10 ppm). The iron tool was fixed in a rotating holder, its circular and axial movements along the vertical axis improved the removing of particles from the working zone and the liquid N_2 was constantly introduced by hand into the device. After machining the particles were garnered by magnets, then dried and cleaned in ethanol.

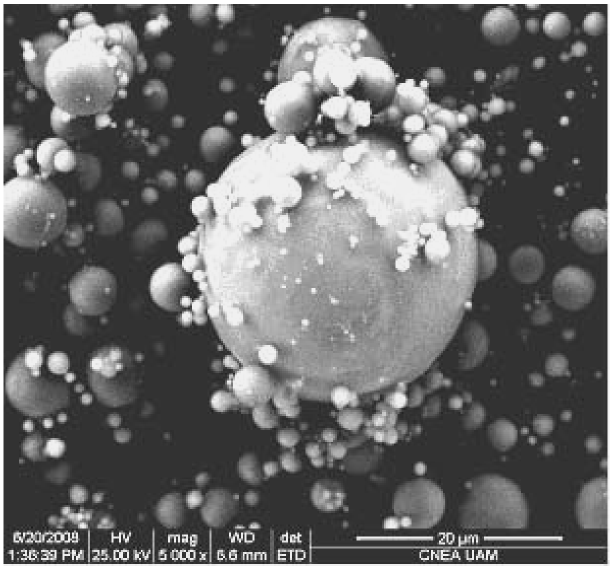

Fig. 3. SEM micrograph or 25 A, 3072 µs particles.

The samples preparation for TEM observation was made as follows: a Cu grid was covered with an amorphous carbon layer, the particles were introduced in an Ependorf recipient, mixed with ethanol and separated by sonication. A small drop of the mix containing the suspended particles was deposited on the grids and laid on the carbon film. TEM observations were performed in a Philips EM 300 operated at 100 kV and in a CM 200 Philips at 160 kV with an EDAX EDS device.

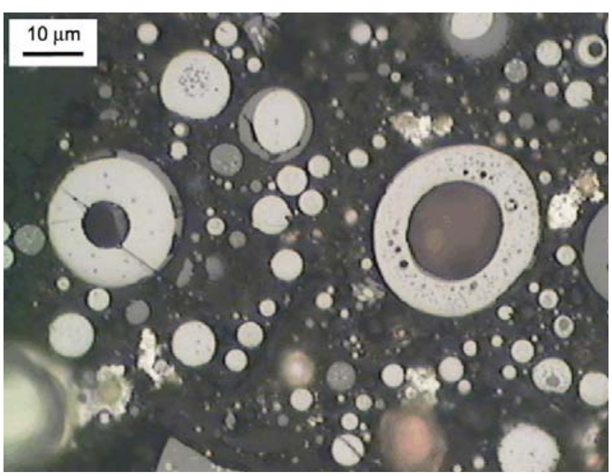

Fig. 4. Optical metalography of 25 A, 3072µs particles.

SEM observations were achieved in a FEI equipment. To observe the interior of particles they were immersed in epoxy and polished in order to reach the sank spheres and observe their bulk.

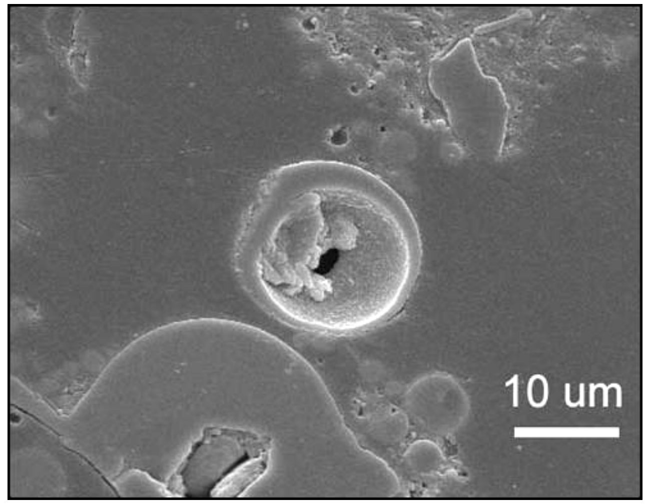

Fig. 5. SEM cross section of 25 A, 3072 µs sample.

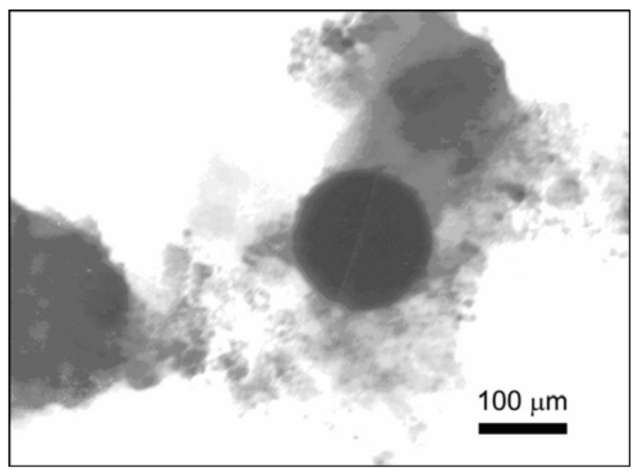

Fig. 6. TEM of 25 A, 3072 µs particles.

RESULTS AND DISCUSSION

In both cases, 9.5 A and 25 A, as a result of this process in liquid N_2 we have got a scarce particles mass. Microscopic spheres are clearly seen after the EDM process by optical and SEM micrographs, Figs. 2 to 5. Figs. 2 and 3 display the external view of particles meanwhile Figs. 4 and 5 are micrographs showing the cross section of hollowed and not hollowed spheres.

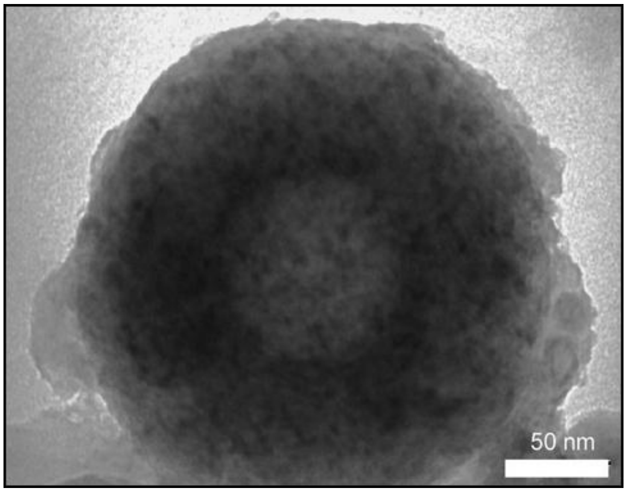

Fig. 7. TEM of 9.5 A, 3072 µs particle.

The TEM micrographs in Figs. 6 and 7 show a general view of particles. The EDS generated in TEM observations have not indicated the presence of N_2 in both group of particles, see Fig. 8. The electron diffraction pattern, not shown, indicated the non amorphous state of this material.

We made a statistical distribution of particles size of both electric currents and the same time t_0, see Figs. 9 to 11.

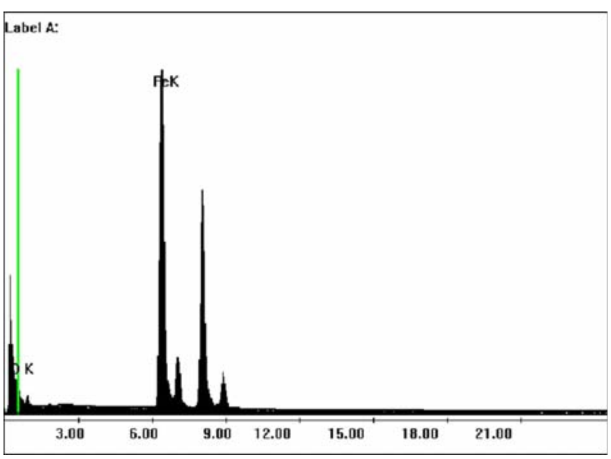

Fig. 8. EDS of 25 A, 3072 µs.

The particles size histograms exhibit a monomodal type, in both cases they are concentrated at 12 nm diameter. The 9.5 A distribution, nearly of 800 particles, do not shows microscopic ones and the 25 A distribution of 1500 particles indicates a small amount of microscopic, larger than 1 µm.

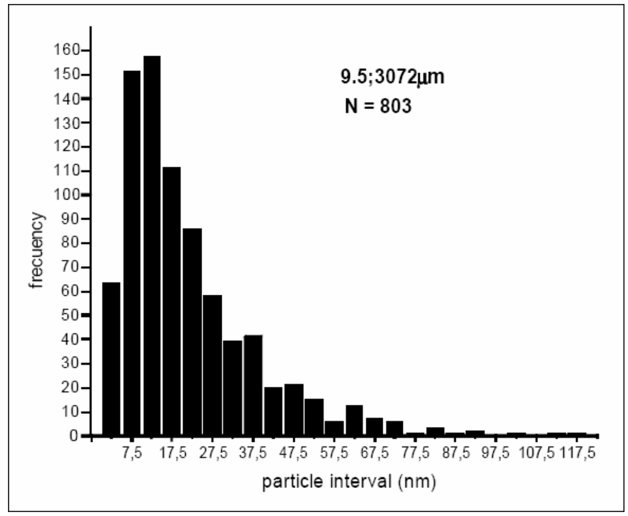

Fig. 9. Distribution of 9.5 A, 3072 µs particles.

Close analysis of spheres surface at high magnification presents a polygonal form. By the way microscopic continuous solid spheres are the most common shape obtained by electro-erosioning iron with kerosene as dielectric, [1]. When water is the dielectric the hollowed spheres are predominant [7]. In the case of the UMo electro-erosioned in water, [9] both hollowed and non hollowed spheres are present. TEM observations of particles obtained using kerosene as dielectric [1] showed only continuous spheres containing carbides as result of the interaction between iron and the kerosene [9]. The distribution of particle size shown in this work indicates that nanoscopic particles are dominant in this process with liquid nitrogen as dielectric, in opposite of what we have found in [9] where, besides nanoscopic, we found microscopic particles or in laser and plasma ablation processes where we have detected both nano and micro particles, [11,12]. Similar results were found with the materials produced by arc melting oven, [13]. Berkowitz [5] suggested the formation of micro particles as result of the expulsed fussed liquid and the nano particles from the metal vapour during EDM. In our case we think that one possible explanation of the main presence of nanoparticles is that the electric energy is not enough to fuse larger amount of liquid metal because the bulk material is at 73 K and probably the gas bubble is small and its explosion might not be intense enough to allow the expulsion of the liquid metal, as occurs when EDM processing is performed at room temperature; this was seen itn the 25 A distribution which has more micromparticles than the 9.5 A one.

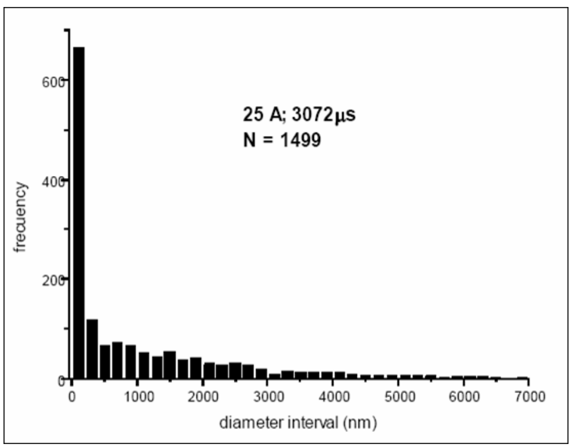

Fig. 10. Distribution of all 25A, 3072 µs particles.

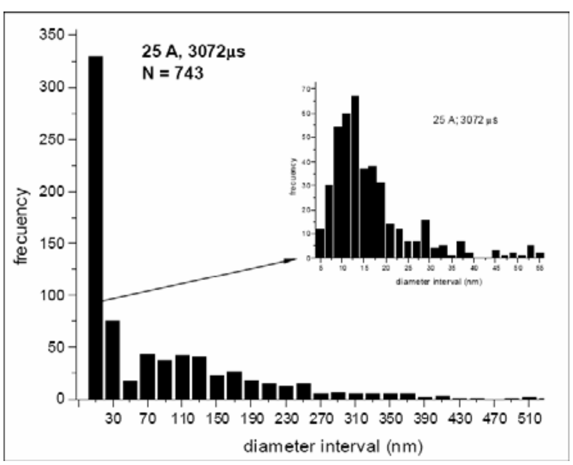

Fig. 11. Distribution of minute particles of 25 A, 3072 µs.

CONCLUSIONS

Mainly nanoscopic spherical material was produced by a commercial EDM electro-erosioning iron in liquid N_2. Nitrides were not found. The amount of the obtained material using liquid N_2 was lower than employing other dielectrics. The measured particles present a monomodal distribution, indicating that particles might be produced preferentially by evaporation. The electron diffraction patterns do not show any amorphous state and TEM observations showed that some particles could be hollowed. This process could be used in order to produce very small particles with different compositions, sizes and physico-chemical properties depending on the dielectric, material to be ablationed and EDM parameters. This simple method could be used to produce small particles for many applications like sintering, alloys, medicine, etc.

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