

# Morphology and Phase Composition of Particles Produced by Electro-Discharge-Machining of Iron

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Abstract. Towards producing metallic particles of controlled size and spherical shape, which are of technological importance, we have collected in the filters of an electro-discharge-machine (EDM) the material ejected from the surface of EDM iron pieces. The conditions of machining were varied for kerosene and water as dielectrics, using a discharge current of 25 A and duration times of 16 and 3072  $\mu$ s for kerosene and of 32, 384 and 768  $\mu$ s for water, respectively. Scanning electron microscopy was used to assess the effect of the time of discharge on the size of the particles. Mössbauer spectroscopy and X-ray diffraction revealed that for kerosene EDM particles only cementite-like carbides of diverse stoichiometry were formed. While no oxide was found for kerosene spheres, the analyses showed that besides the main fraction of  $\alpha$ -Fe, a small percentage of wüstite (and traces of hematite for the 384  $\mu$ s sample) formed on the water EDM ones.

Key words: electro-discharge-machine, Mössbauer spectroscopy, X-ray diffraction.

## 1. Introduction

Electro-discharge-machining (EDM) is a technique used to cut or perforate electrical conductors by means of electric discharges [1, 2]. The sparks propagate across a dielectric medium and produce structural and compositional modifications on the electrodes. The energy density is very high and induces elevated temperatures at the surfaces, which lead to melting and vaporization of the portion of the metal involved in the interaction. In previous works we have studied the modifications induced on the surface by the treatment using kerosene as dielectric [3–5]. We observed spherical craters left by the removed material, which suggested that the expelled substance might imitate this shape.

In this work we report the characterization of the ejected particles produced by EDM on iron tools using kerosene and water as dielectrics and different discharge conditions.

## 2. Experimental

The tool and masterpiece (with an axial hole) were machined of cylindrical shape out of a Goodfellow (FE 007970) piece of pure ARMCO iron with impurities Mn < 800, C < 200, and S < 150 ppm. The electro-discharge-machine was made by CT Electromecánica, Argentina. Two dielectrics were used: pure water of  $10^{-6} \Omega^{-1} \text{ cm}^{-1}$  and aeronautical JP1 kerosene. The electric current discharge pulse was rectangular of 25 A, and of duration times of 16 and 3072  $\mu$ s for kerosene and of 32, 384 and 768  $\mu$ s for water, respectively. The material expelled by the EDM process was accumulated in a device specially designed to collect the particles. The particles were then washed, cleaned and dried with alcohol and analyzed by stereoscopic optical microscopy, scanning electron microscopy (SEM), metallographic microscopy, X-ray diffraction and Mössbauer spectroscopy.

The metallographic observations were performed on particles embedded in an epoxy resin, polished with diamond paste, and chemically attacked with Nital 2 (2 vol.% nitric acid in ethyl alcohol). Mössbauer spectra were taken in transmission geometry in a constant acceleration spectrometer of 512 channels. The data were fitted to Lorentzian line-shapes with a non-linear least-squares program with constraints. Isomer shifts are referred to  $\alpha$ -Fe at room temperature. Observations in a Philips 500 SEM were made on gold-covered samples adhered to a bonded carbon ribbon. The resin-embedded samples were also used for X-ray diffraction (XRD) analyses carried out in a Philips PW1170 diffractometer with Co K $\alpha$  radiation.

#### 3. Results

Stereo-microscopic observations showed spherical particles of various sizes. Figure 1 displays the SEM micrographs of kerosene electro-sparked particles with almost perfect spherical shape whose surface texture can be also seen. Particles obtained after EDM in water are shown in Figure 2.



Figure 1. SEM micrographs at  $170 \times$  of kerosene EDM particles with sparks of 25 A,  $3072 \mu$ s.

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*Figure 2.* SEM micrographs at  $100 \times$  of water EDM particles obtained with sparks of 25 A, 768  $\mu$ s.



*Figure 3.* Metallographic micrographs at  $700 \times$  (left) and  $800 \times$  (right) of kerosene EDM spherical particles obtained with sparks of 25 A, 3072  $\mu$ s.

The particle diameters were measured from digitalized SEM pictures. Their average values are the following. For kerosene EDM particles the diameters for 16 and 3072  $\mu$ s sparks were  $26 \pm 17 \mu$ m, and  $25 \pm 28 \mu$ m, respectively. For water EDM particles the diameters were  $11 \pm 5 \mu$ m,  $17 \pm 8 \mu$ m and  $33 \pm 23 \mu$ m for sparks of 32, 384, and 768  $\mu$ s, respectively. The metallographic observations on the kerosene 3072  $\mu$ s EDM particles are shown in Figure 3.

The XRD patterns are shown in Figure 4. Figure 5 shows the Mössbauer spectra of the spheres produced in kerosene and water. The phases characterized by XRD and Mössbauer spectroscopy are mainly carbides of different stoichiometry – from  $Fe_2C$  to cementite – for kerosene EDM particles, and ferrite and a small percentage of wüstite and hematite for water EDM particles. The results of the fittings of the Mössbauer spectra are shown in Table I.



*Figure 4.* Co K $\alpha$  X-ray diffraction patterns of 25 A EDM samples in water with spark times of (a) 32  $\mu$ s, (b) 348  $\mu$ s, and (c) 768  $\mu$ s, and in kerosene with spark times of (d) 16  $\mu$ s, and (e) 3072  $\mu$ s. The bars at the top indicate the line positions characteristic of the iron phases indicated on the right.

# 4. Discussion

Previous works on particles observed after EDM of Cr, Fe, Al and Sb pieces, showed that their size decreased with the temperature achieved at the surface of the metals [6]. The present results do not exhibit a dependence of the kerosene EDM particles size on the spark duration time and display only a slight dependence for



*Figure 5.* Mössbauer spectra of water EDM particles obtained with sparks of (a) 32  $\mu$ s, (b) 348  $\mu$ s, and (c) 768  $\mu$ s. Mössbauer spectra of kerosene EDM spheres obtained with sparks of (d) 16  $\mu$ s and (e) 3072  $\mu$ s. The fine solid lines show the contributions of the phases indicated in Table I to each spectrum.

the water EDM samples – assuming that the temperatures achieved are proportional to the duration of the sparks.

For kerosene EDM particles, we found both by X-ray and Mössbauer spectroscopy that carbides of different stoichiometry – from Fe<sub>2</sub>C up to Fe<sub>3</sub>C – have formed. Unlike previous observations of EDM surfaces in kerosene [5], no drastic change is observed for such diverse times as 16 and 3072  $\mu$ s. In the case of the products formed on the surface of EDM iron samples using copper tools, both with low- and high-energy sparks a manifest amount of austenite was formed [3]. In the present experiment no austenite has been detected.

	H (kOe)	$\Delta$ (mm/s)	δ (mm/s)	2ε (mm/s)	Relative fraction (%)
			H <sub>2</sub> O		
32 µs					
α-Fe	$330\pm1$		$0.00\pm0.01$	$0.00\pm0.01$	$80\pm2$
wüstite		$0.44\pm0.07$	$0.80\pm0.03$		$20\pm4$
		$0.95\pm0.08$	$0.90\pm0.04$		
384 µs					
α-Fe	$330\pm1$		$0.00\pm0.01$	$0.00\pm0.01$	$80 \pm 3$
hematite	$512\pm4$		$0.35\pm0.06$	$-0.22\pm0.11$	$5\pm 2$
wüstite		$0.44\pm0.08$	$0.79\pm0.05$		$14 \pm 3$
		$0.94\pm0.05$	$0.91\pm0.06$		
768 µs					
α-Fe	$330\pm1$		$0.00\pm0.01$	$0.00\pm0.01$	$90 \pm 3$
wüstite		$0.46\pm0.09$	$0.81\pm0.04$		$10 \pm 2$
		$0.93\pm0.09$	$0.88\pm0.07$		
			kerosene		
16 µs					
	$196\pm2$		$0.20\pm0.01$	$0.03\pm0.03$	$33\pm8$
(a)	$220\pm2$		$0.21\pm0.02$	$-0.05\pm0.03$	$28\pm8$
	$272\pm2$		$0.22\pm0.03$	$-0.05\pm0.05$	$12 \pm 4$
α-Fe	$330\pm3$		$0.00\pm0.04$	$0.00\pm0.08$	$4\pm3$
(b)		$0.86\pm0.04$	$0.21\pm0.03$		$23\pm5$
3072 µs					
(a)	$199\pm3$		$0.20\pm0.04$	$0.02\pm0.03$	$72\pm3$
α-Fe	$332\pm3$		$0.01\pm0.02$	$0.00\pm0.02$	$18\pm2$
(b)		$0.72\pm0.03$	$0.18\pm0.03$		$10 \pm 1$

*Table I.* Mössbauer parameters obtained after fitting the spectra of Figure 5 as described in the text

Isomer shifts are referred to  $\alpha$ -Fe at room temperature. The left column shows the assignments according to the values reported in the literature: (a) values belonging to non-stoichiometric carbides ranging from Fe<sub>2</sub>C to Fe<sub>3</sub>C, (b) superparamagnetic carbides.

The EDM treatment using water as dielectric produced only  $\alpha$ -Fe and a small percentage of iron oxides (mainly wüstite). Only in the case of the 384  $\mu$ s sample some hematite was found, which may have formed after the treatment itself.

Metallography of kerosene EDM particles revealed a Wiedmästaten-like microstructure in all the analyzed balls. The observed needles cannot belong to martensite because this phase was not detected by any of the other characterization techniques used. The microstructure of water EDM particles is completely different from the kerosene ones; the metallographies taken with Nital 2, show only the characteristic form of iron oxides. Some particles exhibit holes in their centers. They might have formed by segregation during the cooling stage, or brought about by gases occluded during the process.

# 5. Conclusions

The current results show that EDM is a promising method for obtaining metallic particles of spherical shape. The carbides obtained in kerosene may find use in some industrial applications. EDM in water seems an advantageous and economic way to produce particles made up of almost the pure initial material. More experiments are in progress to control the size of the particles and the products obtained (trying to minimize the amount of surface oxides for water EDM particles) by varying the different parameters of the EDM process.

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