

## Microscopic spheroidal particles obtained by laser cutting

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Metallic spherical microparticles are needed for a wide range of applications in powder metallurgy. They can be produced by several well-known methods [1]. Within a research program aimed at the fabrication of uranium-molybdenum particles for nuclear fuels for research reactors [2], we have already investigated the use of the electron discharge process to obtain spherical iron particles using kerosene as dielectric [3] and also applied this method to the production of U-Mo spherical particles using pure water as dielectric [4].

Laser ablation [5] is a process currently used to cut and also to modify the characteristics of material's surfaces [6–9]. As far as we know, the nature of the removed material from the cutting zone has not been studied yet.

In this work we study the characteristics of the particles ejected from the cutting zone of a SAE 1010 steel while using a CO<sub>2</sub> gaseous laser with the purpose of determining the feasibility to use this technique in the production of microscopic particles for nuclear applications [2] as well as for other scientific and technological uses [1].

A 1.65 kW CO<sub>2</sub> gaseous laser was tuned to a wavelength of 10.6 μm, 99.9% purity oxygen at 69 KPa was used as contributing gas for cutting. The gas flow was 6.67 × 10<sup>-4</sup> m<sup>3</sup>/s. The laser cutting was carried out in open air on a 6.35 mm thick SAE 1010 hot rolled steel plate. The cutting velocity was 31.75 mm/s. The ejected particles were collected, sieved, classified by size, and then observed by scanning electron microscopy (SEM) and optical microscopy. Some particles were mounted in epoxy resin for metallographic observation. The particles were analyzed by X-ray diffraction using Cu-K<sub>α</sub> radiation. Particles smaller than 45 μm were analyzed by Mössbauer spectroscopy in transmission geometry employing a conventional spectrometer with constant acceleration and 512 channels using a 15 mCi <sup>57</sup>Co-Rh source. Isomer shifts were calibrated against an α-Fe foil at room temperature. The spectra were fitted to Lorentzian line-shapes with a least-squares program with constraints.

Table I shows the mass and size distribution of the particles. Fig. 1 shows that the particles with sizes between 75 and 104 μm exhibit a spherical appearance. The micrograph in Fig. 2 displays some external characteristics of the spheres, like hulls and perforations in the group of particles larger than 104 μm. Fig. 3 shows the internal aspect of particles 75 to 104 μm in diameter. Some of them are hollow and others contain a complex structure with internal minor features. The qualitative results of the X-ray diffraction data are included in Table I. Fig. 4 shows the Mössbauer spectrum of the sample whose particles are smaller than 44 μm. The hyperfine parameters,  $H$  = hyperfine magnetic field,  $d$  = isomer shift referred to α-Fe at room temperature and  $2e$  = quadrupole shift, reveal that the particles are made up of more than 87 ± 3% of magnetite ( $H_1 = 49.6 \pm 0.1$  T,  $H_2 = 45.8 \pm 0.2$  T,  $d_1 = 0.33 \pm 0.01$  mm/s,  $d_2 = 0.67 \pm 0.01$  mm/s,  $2e_1 = 2e_2 = 0.00 \pm 0.01$  mm/s). The central region also reveals the presence of 13 ± 3% of a superparamagnetic iron oxide. The X-ray data agree with this assignment.

In general, spherical and non-spherical particles are used in powder metallurgy. The production methods can be classified as mechanical—e.g., milling, grinding, etc.—and melting where particles are formed during cooling—e.g., atomization, rotating electrode [10], electro erosion, etc. Nuclear fuels employ non-spherical particles as uranium oxides or uranium silicides, but in

TABLE I Particles divided in groups after sieving at the indicated mesh, corresponding sizes, average masses, and phases detected by X-ray diffraction and Mössbauer spectroscopy. The phases found in greater proportions are shown in bold type

Group	Mesh	Size (μm)	Mass (g)	Detected phases
1	–325	<45	1.26	<b>Fe<sub>3</sub>O<sub>4</sub></b> , Fe <sub>2</sub> O <sub>3</sub>
2	325	45–75	6.04	<b>Fe<sub>3</sub>O<sub>4</sub></b> , Fe <sub>2</sub> O <sub>3</sub>
3	200	75–104	6.14	<b>Fe<sub>3</sub>O<sub>4</sub></b> , Fe <sub>2</sub> O <sub>3</sub>
4	+150	>104	69.6	<b>Fe<sub>3</sub>O<sub>4</sub></b> , FeO, α-Fe, Fe <sub>2</sub> O <sub>3</sub>

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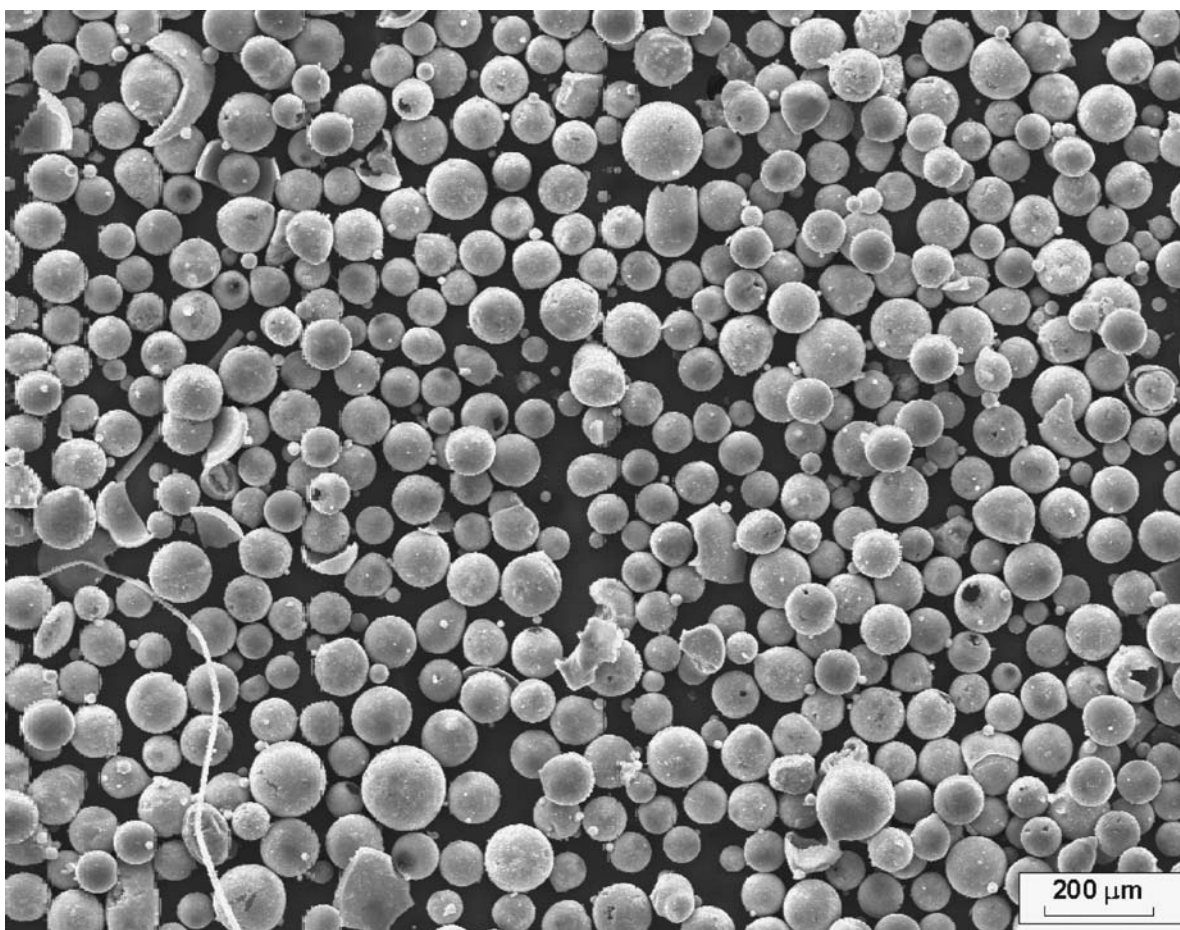


Figure 1 Micrograph showing the spheroidal appearance of particles from 75 to 104  $\mu\text{m}$  in size.

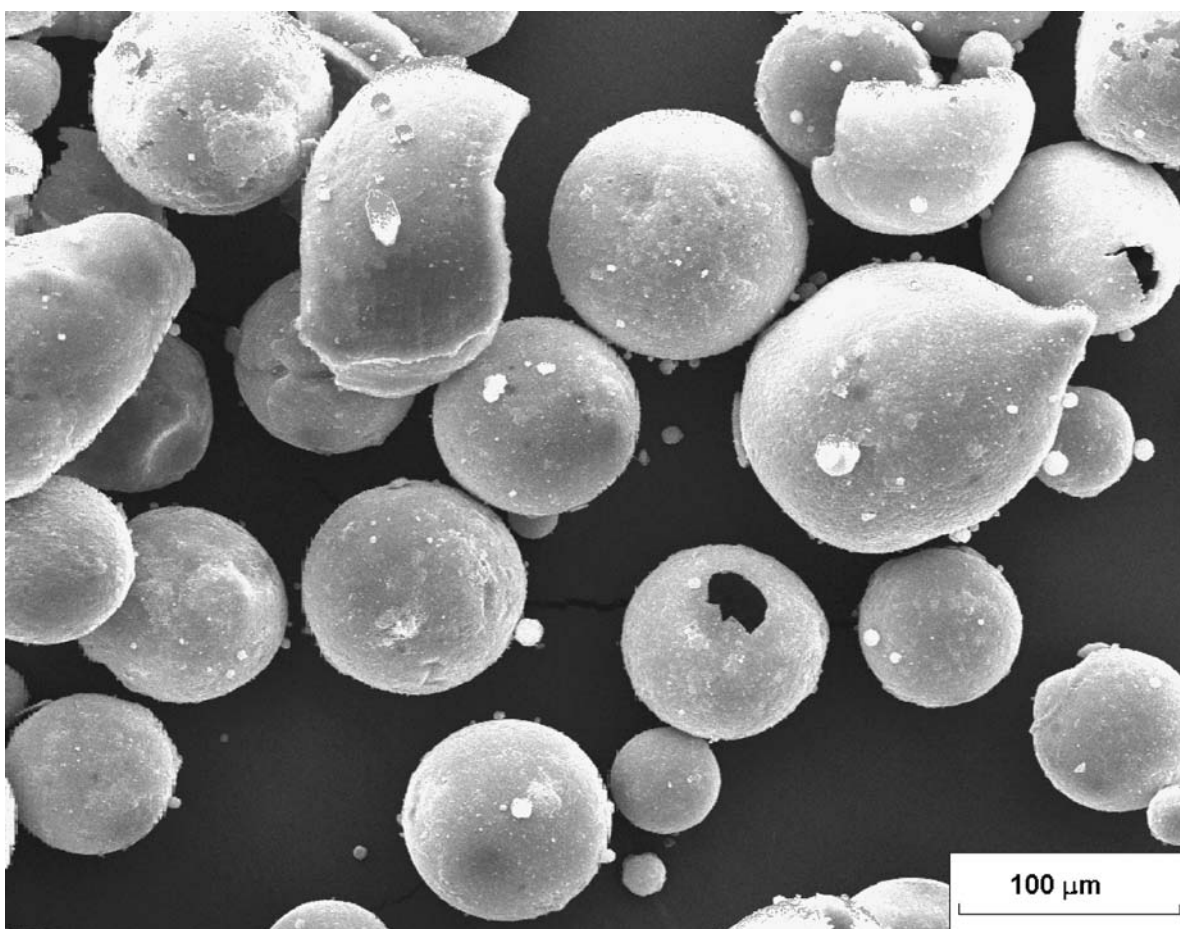


Figure 2 Hull flakes and perforated spheres in particles larger than 104  $\mu\text{m}$ .

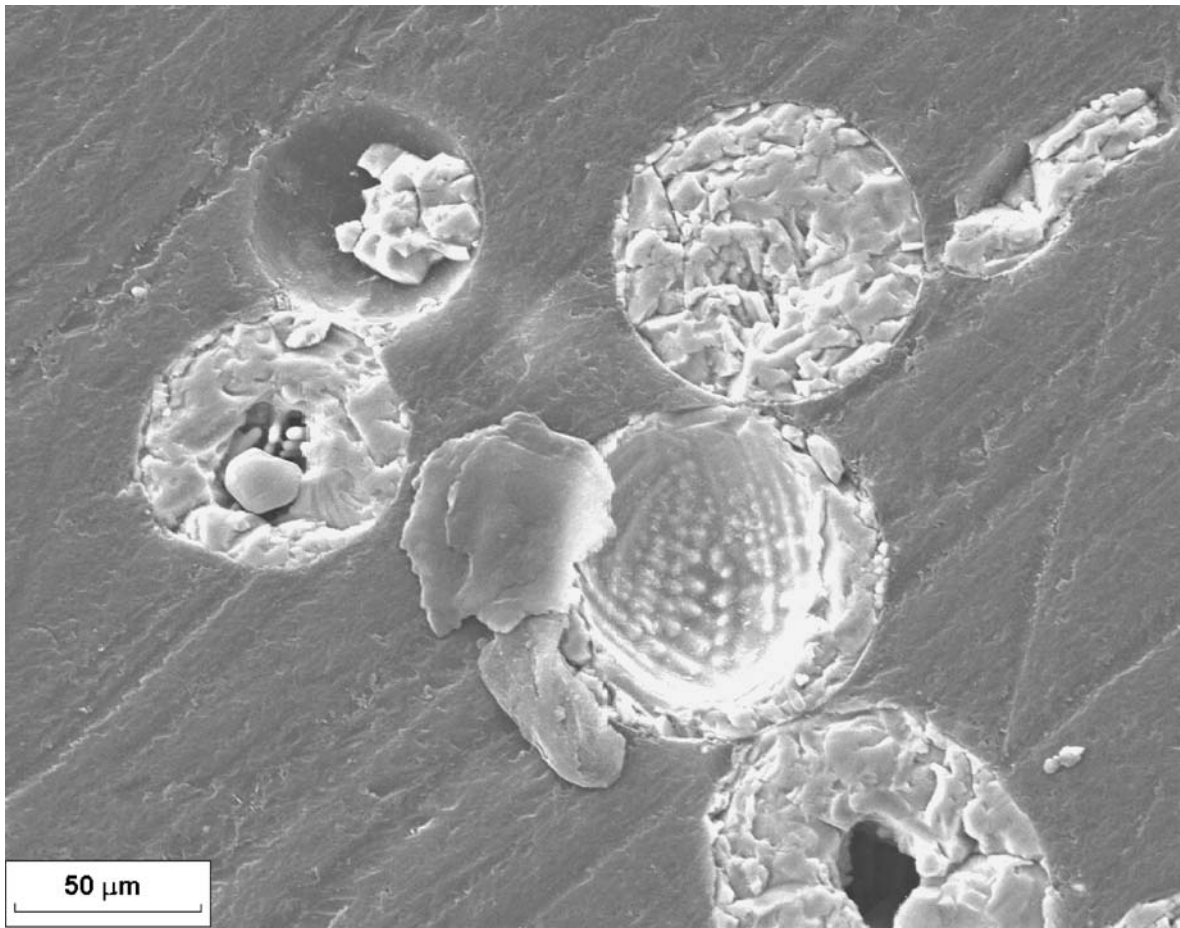


Figure 3 Internal particles look of spheres showing a complex structure in particles of average sizes between 75 and 104  $\mu\text{m}$ .

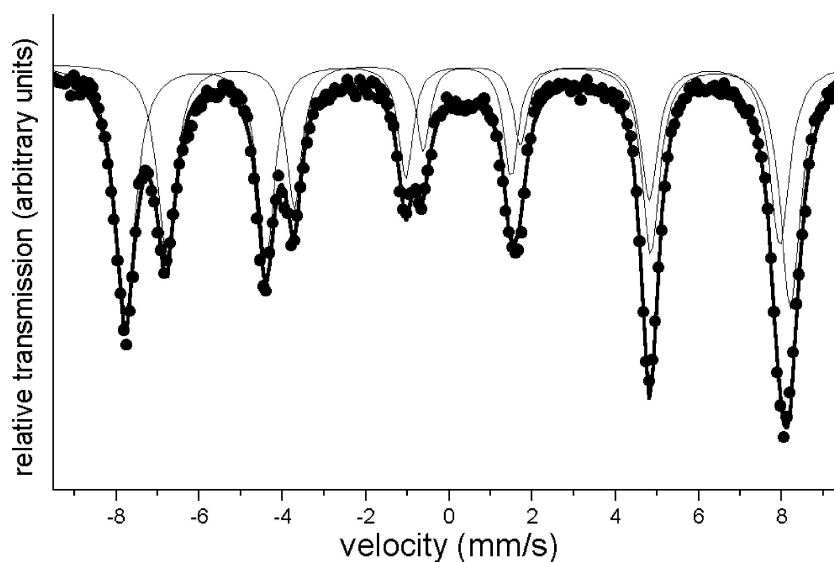


Figure 4 Mössbauer spectra obtained from particles smaller than 44  $\mu\text{m}$ . The solid line is the least-squares fit to Lorentzian line shapes as described in the text. The thinner lines are the two magnetite components belonging to sites A and B of its structure.

recent years alloys of uranium-molybdenum (U-Mo) have been proposed as suitable materials to be used in nuclear fuels for research reactors [2].

Recently, we have produced U-Mo micro particles [11]. The method of hydriding-milling-dehydriding (hmd) that we developed yielded particles of non-spherical shape, while spherical particles of U-Mo were obtained by electroerosion [4].

Simple energy considerations that take into account the surface tension are enough to explain the spherical

geometry of particles obtained during the quenching after their ejection produced by laser ablation. The hot material, liquid or solid, oxidizes easily in the oxygen flow when ejected from the cut zone. It is well known that the oxidized iron structures increase their dimensions by approximately one third with respect to the metal. This may explain the existence of disassembled rinds with or without interior small spheres, and could also explain the observed isolated shreds broken up from spheres.

Samples 1, 2 and 3 do not show indications of FeO or  $\alpha$ -Fe. Only in the larger particles, sample 4, some FeO was formed and a little  $\alpha$ -Fe was retained from the initial material. All the samples have magnetite and hematite. The presence of FeO and  $\alpha$ -Fe in sample 4 could be an indication of the oxidation mechanism that takes place in laser cutting in air when a CO<sub>2</sub> laser is used to cut a SAE 1010 steel. In the larger particles the oxidation is not so fast and some part of the material remains as Fe with incipient FeO.

Power laser cutting is appropriate to produce particles for many purposes, specially if an adequate reservoir with controlled atmosphere is made in which the ejected particles can cool down to room temperature while avoiding contact with air—e.g., in an argon chamber large enough to allow the cooling of the hot ejected particles in their flight. We think that the spheres have specific physical properties depending on their shapes. In particular, the iron-oxidized ones, which are hollow, may serve for purposes of technological importance.

For other applications, the atmosphere used may contain an appropriate gas to purposely encourage the formation of diverse complex compounds. Specifically, the production cost of spherical particles for nuclear fuels might be greatly decreased if the current method—using appropriate parameters—was found to produce the desired shapes and sizes of the particles needed in the nuclear industry.

These preliminary results show that laser cutting can be applied to obtain spherical metallic particles. With regard to the manufacturing of nuclear fuels this process can compete with other production methods.

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