Transmission electron microscopy observation of nanoparticles obtained by cutting power laser

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In recent years the interest in nanosized particles has been increasing continuously. The design of nanostructured materials that possess novel magnetic, optical, electronic, metallurgical and mechanical properties has motivated the development of these materials. Surface and size effects are particularly important in the physical-chemical behavior of ultra fine powders and new applications are now being discovered. For instance the importance of iron-oxide-based materials for applications like catalysis, pigments sensors and magnetic recording media, induced the study of the properties of ultra fine powders of these compounds.

Laser ablation [1, 2] is the basic process connected with metal cutting. Cutting of metals is one of the well known technological laser applications [3–5], being essentially a thermal process. Nanoscopic particles having a narrow size distribution [6] have been observed by exposing micro particles to a pulse laser energy beam above the ablation threshold of the micro particles. Iron nanoparticles were observed by TEM when olivine samples were subjected to pulse laser irradiation, [7]. Silicon nanoparticles were also produced employing a pulse laser [8]. Electron diffraction and TEM observations were carried out on HgCdTe laser deposited films [9]. However, the ejected material produced by cutting lasers has not been yet studied. In a research program aimed at the production of nuclear fuels for research reactors with uranium-molybdenum particles, we initiated a number of works in order to obtain fine and ultra fine particles by power lasers. In this work the first result of the nanoscopic characteristic of the ejected particles coming from the material in the cut zone of a SAE 1010 steel is shown.

We have employed a 1650 W CO_2 laser, 10.6 μm wavelength with oxygen as the cut contributing gas, which is used for the material removing even in open air atmosphere. The cut material was a SAE 1010 steel, 6.35 mm thick, hot rolled with unstripped surface. The particles from the laser made trench in the steel were picked up from the reservoir of material debris, and filtered through a 325 mesh sieve allowing separation of particles smaller than 44 μm. The macroscopic shape of the particles was analyzed by scanning electron microscopy (SEM). TEM and electron diffraction observations were carried out in a CM 200 Philips microscope operated at 200 keV. The sample preparation was the usual to observe particles with TEM: copper grids with a carbon layer and dropping methanol on it with suspended particles. The powder passing through the 325 mesh was also analyzed by X-ray diffraction (XRD) using Cu K_α radiation.

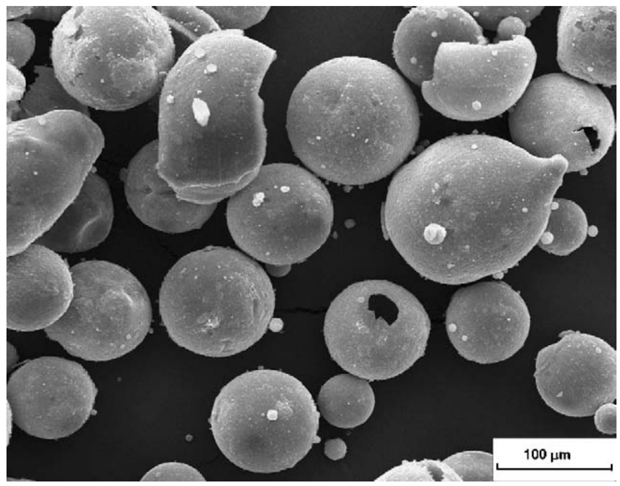

Figure 1 Microscopic Spheroidal particles, between 44 and 75 μm in size. Some balls have hulls and others are perforated.

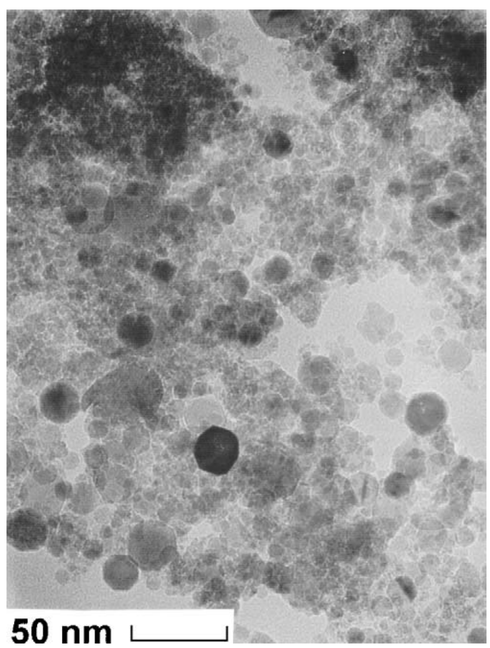

Figure 2 TEM micrograph of the laser ejected nanoscopic particles randomly congregated on the carbon supporting film.

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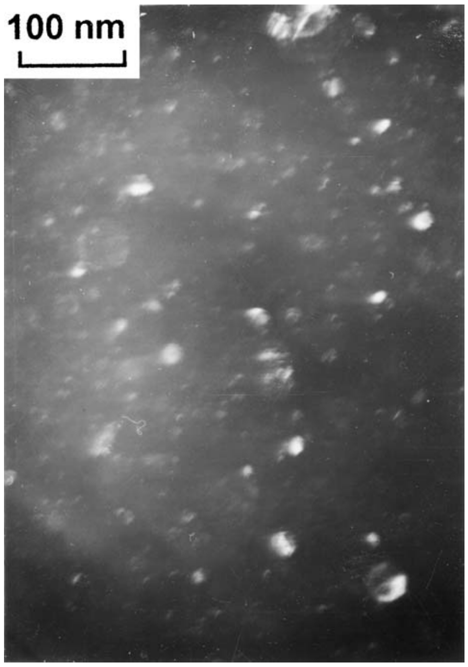

Figure 3 TEM dark field micrograph, showing the crystallite size.

The SEM micrograph in Fig. 1 shows the spheroidal aspect of particles larger than 44 μm and smaller than 75 μm, some hulls and perforated balls may be seen. The TEM micrograph in Fig. 2 indicates the existence of spheroidal nanoparticle clusters on the carbon layer. In the dark field micrograph in Fig. 3 crystallites can be seen whose dimensions are smaller than the particles themselves. The electron diffraction pattern, not shown, and the XRD in Fig. 4 indicate that the particles have a crystalline structure and consist of iron oxides, principally magnetite and hematite. From the TEM micrographs 119 particles were measured and the size distribution is shown in Fig. 5; their mean value is about 20 nm. From dark field TEM micrographs it was also possible to measure the crystallites size with a mean value of about 10 nm.

When a power laser beam impinges on a small surface area of a metal, it raises the local temperature reaching the melting point and forms a weld pool. Pressured gas coming through the nozzle of the apparatus emerges with the laser beam and forces the liquid to be ejected from the pool to the surrounding atmosphere.

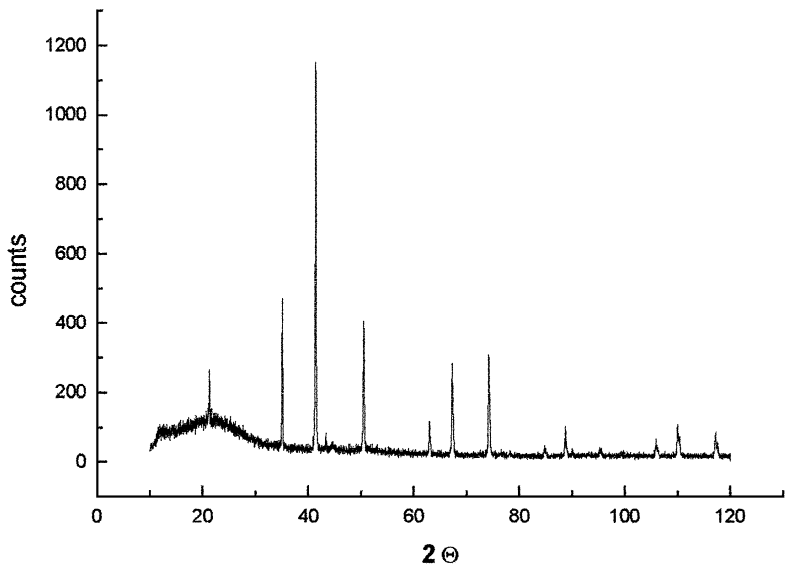

Figure 4 X-ray diffractogram of the powder taken with Cu K_α radiation, the lines correspond to hematite and magnetite.

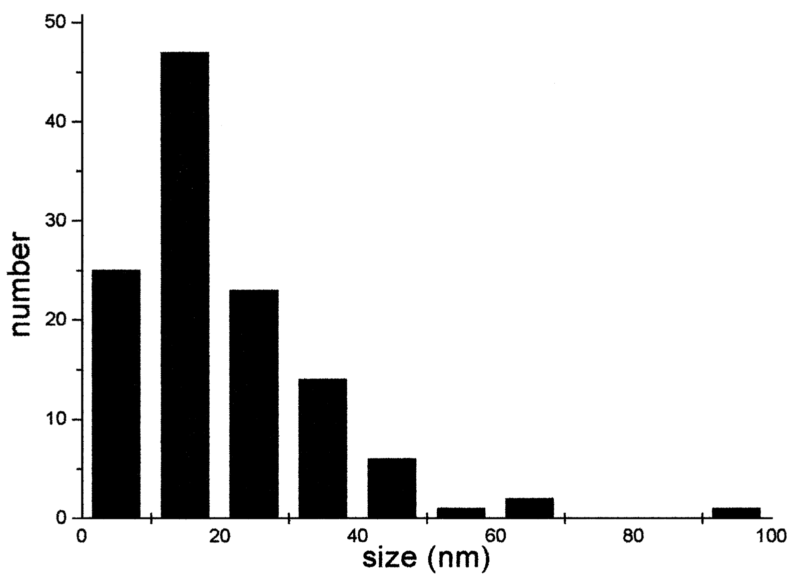

Figure 5 Histogram of the particles size, with a mean value of about 20 nm.

The removed liquid, obeying minimum energy principles, gives spherical shapes with their own micro metric dimensions [10], when flying through the atmosphere, see Fig. 1. The TEM observations demonstrate the existence of spheroidal nanoparticles, see Fig. 2. The dark field information indicates that the crystallites are half the size of the nanoparticles, which could thus be considered as multi crystalline. Electron and X-ray diffraction patterns yield evidence of the presence of iron oxides, such as hematite and magnetite. The oxidation was produced by the interaction with oxygen as a contributing gas, when the hot material is driven out from the cut zone into open air. The shape and size of nanoparticles are similar to those observed in [7]. However, there is a difference in the composition, since in that work only iron was observed in the particles.

The power laser cutting is an interesting procedure to produce nano-particles. Their properties could be of interest for future investigations, especially if oxygen is not used and a reservoir with a controlled atmosphere is employed, in which the ejected particles could cool to room temperature without contact with air. The atmosphere could also be constituted by gases appropriate for the formation of other complex compounds. Work is currently being carried out in order to obtain more details regarding the characterization of these types of materials and to study their properties.

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