GENERATION OF FERROMAGNETIC MICRO- AND NANOPARTICLES BY LASER AND MECHANICAL MILLING METHODS

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Introduction

Dispersive, fine structured or nanostructured materials and nanoparticles are usually produced by complicated and expensive methods. Various ways to produce nanostructured materials have been in usage, among them chemical synthesis, destruction of wires by a strong electric current, spraying of a special liquid, melting, and laser methods such as surface processing, ablation, etc. The application of a laser makes possible to obtain nanostructured materials with the composition close to that of the initial material [1].

Nowadays, for the production of nanocrystalline materials, laser methods – ablation, surface evaporation, laser cutting as well as others – are widely used [2]. These methods can be employed to obtain even very small (3–10 nm) nanoparticles suitable to make qualitative colloids [3, 4].

This article describes the method of laser cutting applied for the generation of small particles when particles of different sizes are formed in the gas flow during the steel cutting process [1]. In addition, it demonstrates that the particles formed in the gas flow can be separated, at their size, using the cyclone and fine synthetic cloth as well as analytical filters [5].

Experimental methods

Laser cutting in the optimal cutting regime of the strips of stainless steel 7C27Mo2 (SANDVIK) of the thickness of 0.2–0.6 mm was used to generate particles. The LIT-100M laser was applied. The original material was processed with 2±0.6 ms Nd:YAG laser pulses having 150±3 Hz frequency and the overall power of up to 250 W. The radiation was focused onto a 0.18–0.25 mm diameter area. To manipulate the laser beam and to transmit it to the cutting zone, the principle of the flying zone was applied [2]. In the experiments, the average power of 100–150 W was used. The average cutting velocity was 100 cm/min. The airflow was directed to the cutting zone applying the conical nozzle whose diameter of opening was 0.8–1.2 mm. The pressure of gas before the nozzle was 0.35–0.6 MPa. A scheme of the laser generation of micro-and nanoparticles is presented in Fig.1. The larger particles at the size of micrometers were deposited in the chamber and the cyclone (2 and 3 in Fig. 1). Such particles were further processed by the planetary mill Pulverisette 6, with the balls of wolfram carbide of 15 mm and steel balls of 5 mm.

The phase composition of the initial material and that of the particles was determined using Mössbauer spectroscopy. Mössbauer spectra were measured in the transmission regime at room temperature using ^{57}Co(Rh) source. To find relative contributions of different iron compounds (α-Fe(Cr), magnetic Fe_3O_4) and $(FeCr)_3O_4$ phase or superparamagnetic particles, the corresponding sub-spectra were fitted to the experimental spectra.

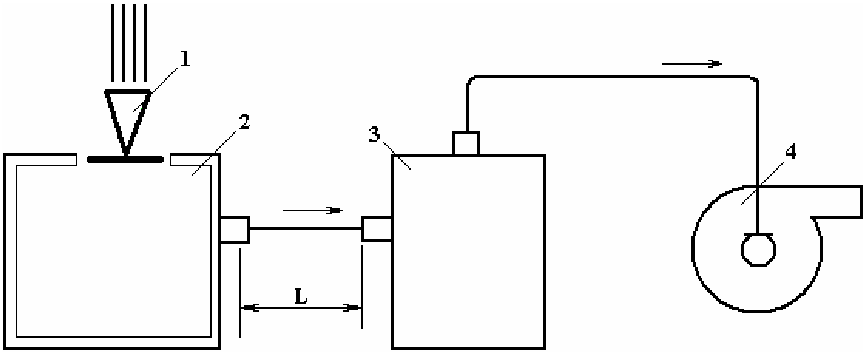

Fig. 1. Scheme of laser generation of micro- and nanoparticles: 1– laser beam, 2 – chamber, 3 – cyclone, 4 – pump, L – tube length

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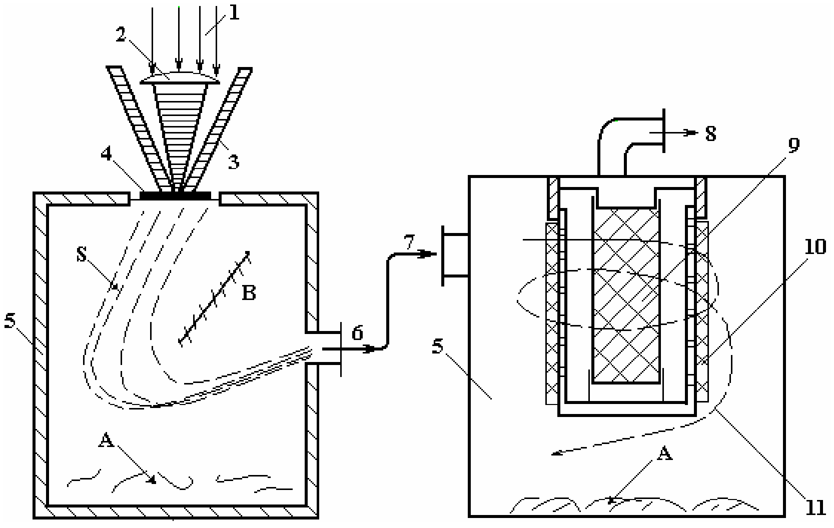
Results and discussion

The generation of micro- and nanoparticles using laser cutting takes place when particles are formed from the melt in the cut slit in the gas flow.

Optimal cutting regime and separation of small particles

The largest effect of laser cutting is obtained at the optimal power of the laser and the pressure of gas before the nozzle. The particles of micro-nanosizes formed in the slit oxidize in the gas flow when they move away from the cutting zone [1, 6]. The particles of up to 100 μm are deposited in the chamber and the cyclone (Fig. 1), while the particles of 1μm-10 nm are found in the cascade filters [5], which can used for the production of tribosuspensions.

A more detailed picture of the processes in the chamber and the cyclone is shown in Fig. 2. The heavier particles, which make up about 40–45% of the generated particle mass, are deposited in the chamber (Fig. 2,*a*). The distance between the chamber and the cyclone may be changed, which influences the transfer of particles to the cyclone. For the distance (tube length) of 0.5 m the flow of particles in the cyclone was spiral-like assisting the deposition of other 20–25% of the heavier particles on the bottom and the buffer filter of the cyclone. The second cascade filter (9, Fig. 2,*b*) is synthetic and traps 10–15% of the particles smaller than 10 μm. The particles found in the chamber and the cyclone are of 10 μm – 100 μm and can be further milled to decrease their dimensions.



a *b*

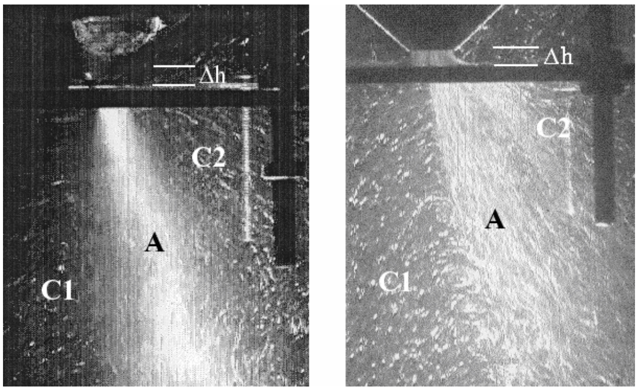
Fig. 2. Distribution of particles in the airflow receding from the destruction zone of material: 1 – laser beam, 2 – lens, 3 – nozzle, 4 – destructed material, 5 – chamber(a) and cyclone (b), 6 – outlet flow, 7 – inlet flow, 8 – outlet flow of small particles, 9 – filter of cascade II, 10 – buffer filter, 11 – whirlwind flow of particles, A – site of deposition of large particles, B – reflector, S – flow of particles

Modeling of air-particle flow

The formation of particles, their size and further deposition depend both on the processes of laser cutting that are influenced by the changes in the laser power, dynamics of the technological gas flow and nozzle parameters and on the distance between the particles flown in the system.

The dynamics of the gas flow is essential for laser cutting. Therefore, the characteristics (velocity, pressure) of the gas flow in the opening of the nozzle, between the nozzle and the surface of the destroyed material, and on the other side of the slit were studied [7]. The optimal cutting regime is achieved when only a minimal amount of erosion products remains on the cut edge. The optimal regime is obtained at the gas pressure of 3.5–6.0 bars and the sufficient power of the laser beam.

The flow of a technological gas in conical nozzles and below the slit of the cut was modeled. The experiments with the nozzles of real dimensions using flows with the glycerin and water particles were made (Fig. 3) [8]. In the ring nozzles, it was possible to form the flow with the highest average velocity in the opening of the nozzles, therefore those nozzles could be most effective for working conditions. The outlet opening of the ring nozzle between d= 8mm and d_0= 10 mm formed the ring-like gas flow (Fig. 3,*a*). The comparison of flows shown in Fig. 3,*a* and *b* allowed us to state that in ring flow the particles below the slit move visually faster compared with those in the cylindrical flow. The area of the flow (A) is more outspread. (Fig. 3,*b*). The parameters of the gas flow have a bigger effect on the quality of laser cutting because the liquid metal from the cutting slit is removed by the effective gas flow formed by the nozzle.



a *b*

Fig. 3. Gas flow visualization behind the obstacle: a) ring flow (flow velocity $U_o > 100$ m/s, and b) cylindrical flow (flow velocity $U_o \sim 50$ m/s), ***A*** *– the main jet,* ***C1, C2*** *– recirculation regions, the inner angle of conical nozzles $2\alpha = 70^0$, $d_0 = 10$ mm, the slot $d_1 = 1.2$ mm, $\Delta h = 2.0$ mm*

In another study, the airflow in the tube of the 30 mm in diameter was modeled, with the distance between the cutting place and the particle collection place being 0.5, 1.0 and 3.0 m. The scheme used for simulation is shown in Fig. 1. The results of the simulation in case of the 3.0m length tube, with the inlet velocity of 80m/s, are presented in Fig. 4. The finite volume method was used for the numerical solution of two-dimensional differential equations which can be presented in a generalized form [9, 10]:

$$\frac{\partial}{\partial t}(\rho\Phi) + \mathrm{div}\left(\rho\vec{\mathbf{V}}\Phi - \Gamma_\Phi\,\mathbf{grad}\,\Phi\right) = S_\Phi\,, \tag{1}$$

where t is time, s, ρ is the density, kg/m^3, Φ is the dependent variable (Φ = 1, u, v, k, ε are, respectively, continuity equations, velocity components in x and y directions, m/s, kinetic energy of turbulence and its dissipation rate); **V** is the velocity vector, m/s, Γ_Φ is the exchange coefficient, S_Φ is the source term for variable Φ, $k = (u^2 + v^2)/2$ in the case of two dimensions. The coefficient Γ is the sum of coefficients of laminar and turbulent exchange:

$$\Gamma_\phi = \Gamma_{l\phi} + \Gamma_{t\phi},, \tag{2}$$

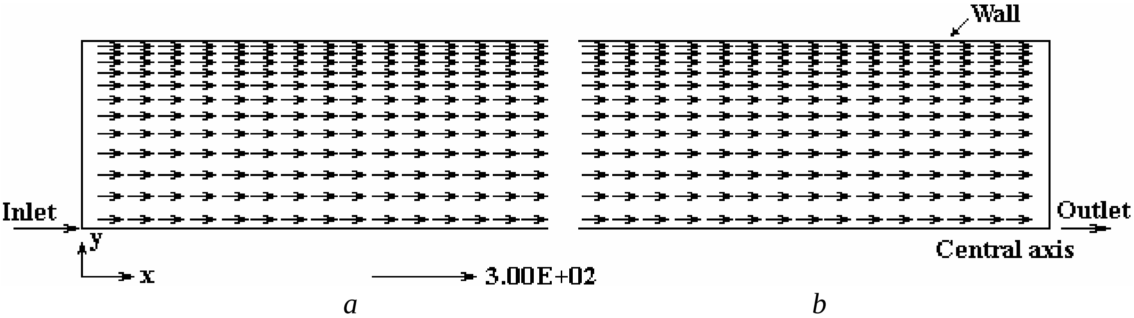
which in PHOENICS CFD [9] is expressed using density ρ and laminar and turbulent viscosity, ν_l and ν_t, by the expression

$$\Gamma_\phi = \rho\,(\nu_{l\phi} + \nu_{t\phi}). \tag{3}$$

The additional turbulent viscosity [9] is:

$$\nu_t = 0.09\,k^2/\varepsilon. \tag{4}$$

The partial differential equations for k and ε are solved using a standard ***k-e*** turbulent model. The kinetic turbulent energy is about 70 at the wall and 52 in the center of the tube at the inlet (Fig. 4). It decreases to the values of 43 and 15 along the whole tube length (3 m).



a *b*

Fig. 4. Velocity vectors: a) at the inlet of the tube (tube section length is 0.5 m (0 – 0.5 m)); b) at the outlet (2.5–3.0 m) of the tube; the tube length is 3.0 m, diameter – 0.03 m, inlet velocity is 80.0 m/s; velocity vectors scale is 300.0 m/s (velocity profile at the outlet: v_{min}=51.5, v_{max}=92.7 m/s)

Modification of materials by other methods

When using laser cutting for the generation of micro- and nanoparticles, only 20% of the particles were collected in the filter cascades. The rest 80% of the collected particles were larger than 1 μm. To increase the production of nanoparticles those particles were milled using the planetary ball mill [11]. The duration of milling was up to 5 hours. For the milling, 10 and 25 wt. % of the particles of such soft materials as Zn and Sn (which can be advantageous for tribological purposes) were added to those generated by laser cutting.

Studies of Mössbauer spectra of collected and milled particles

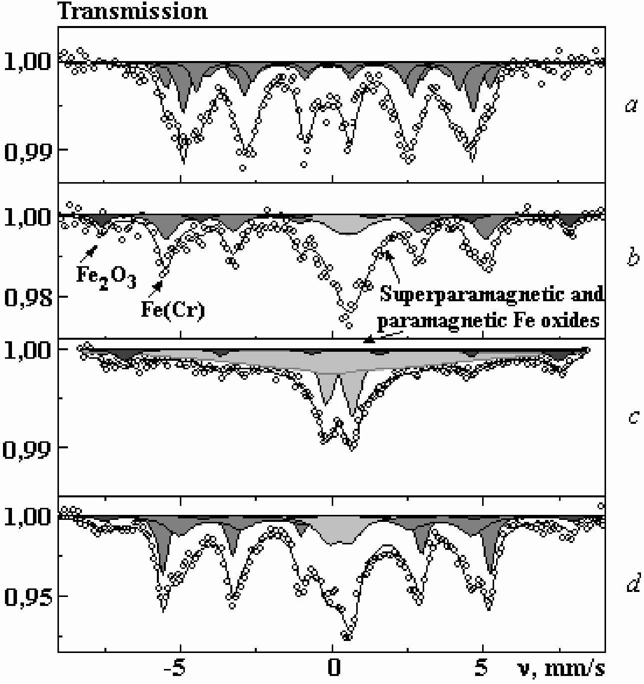


Fig. 5. Mössbauer spectra of: a) initial steel, b) particles produced by laser cutting (cascade I of filters), c) particles produced by laser cutting (cascade of filters IV), d) milled particles with 10% wt Zn

Mössbauer studies revealed changes in the composition of the material collected in different filter cascades (Fig. 5,*b,c*). In cascade I, the material usually contained a large amount of the initial material (Fe, Fe(Cr)). As in the following cascades, with the increase of the degree of oxidation, the material turns to be composed mainly of magnetite and becomes superparamagnetic [12]. In the Mössbauer spectra of the material collected in cascade IV, the superparamagnetic contribution of nanosized particles (<10 nm) was evident. After milling the particles produced by laser cutting, together with those of Zn and Sn, the changes in Mössbauer spectra were observed (Fig. 5,*d*). The changes indicated chemical alloying and the interaction between the particles of different chemical compositions.

Conclusions

The method of the laser generation of particles, laser cutting suitable for the generation of particles of different dimensions and chemical compositions, close to those of the initial material, has been proposed. The parameters of the laser beam and the gas flow, which makes it possible to generate micro- and nanoparticles, have been determined. The particle modification with aluminium and zinc by milling has been performed. The modelling data have confirmed that the most effective generation of small particles from the liquid metal state in the cutting slit is when using nozzles that form ring flows.

Acknowledgements

This research was funded by the Research Council of Lithuania, AUT-11/2010.

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Received 01.11.10

Summary

The generation of micro- and nanoparticles using laser cutting of steel strips of the thickness of 0.2–0.6 mm and the separation of particles according to their size were studied. Under optimal cutting conditions (laser power, gas pressure before the nozzle), the cutting quality is good when a small amount of fully oxidized erosion products at the cut edge of a slit is obtained. The study showed that the cascade filter system can be used to separate the particles up to nanometer dimensions. The simulation of the gas flow can be used to create optimal conditions for collecting generated particles of the required dimensions. The modelling data confirmed that small particles from the liquid metal state in the cut slit are generated most effectively using nozzles forming ring flows. The larger particles modification with aluminium and zinc by milling was performed.