Working medium supply systems for laser plasma generators

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Abstract. Despite large number of papers devoted to laser-induced plasma, there are very few studies on working medium supply systems application in laser plasma generators. At the same time, a number of technologies require stable, ultra-fine working medium supply; high reliability and lifetime; compactness; low energy consumption; ability to change medium; possibility of additional impact on working medium and plasma plume. As a result of the analysis of solid and liquid working media supply and dosing systems, it becomes obvious that combination of their advantages and partial elimination of disadvantages is possible using systems in which transfer, supply and dosing are carried out in liquid state, and immediately before the impact, there is an increase in their viscosity or solidification due to physical or chemical processes. There are no practical restrictions for specific parameters matching (single dose size, dosing stability, frequency of sampling, integral mass flow, lifetime), but number of options satisfying multiple, and often conflicting, requirements simultaneously (including weight and size) is limited.

1. Introduction

Despite large number of publications devoted to laser ablation, there are only few works devoted to working medium (WS) supply systems (SS) for laser plasma generators. At the same time, industrial applications require a careful supply system selection, including: stable discrete, often ultra-small, mass flow rate of a given WM; high reliability; long lifetime; compactness; low energy consumption; ability for switching WM; possibility of additional impacts on WM and gas-plasma flow (GPF). Supply and dosing systems should be considered from the standpoint of WM aggregate state: solid (or solid matrix); liquid (including pastes and gels); combined (with phase transitions). Gas supply systems will be considered here only briefly, mainly related to aerosols. The creation of special WM SS is required for coatings deposition; scale simulators of shock waves and GPF impact; pulsed plasma thrusters, etc.

Laser plasma generation has been considered mainly for solids rather then liquids [1], since high energy density can be reached in a surface layer of the former, and plasma plume containing mainly high speed light particles [2]. Early works declared simple solids supply systems (rotating disk or cylinder) as an advantage for laser thrusters, but this was right for lab modeling only. Applied research has revealed numerous problems with reliable, long-term, exchangeable solid medium supply in industrial laser plasma generators prototypes. According to laser ablation specific energy, medium dosing should be available down to $10^{-5}$ g/J, output $10^{5}$ g/s to 1 g/s with stability better than 5%, and virtually no capacity limits. For lifetime possibly close to lasers source (10^3 hrs for diode lasers), resistance to laser impact would be crucial. Energy efficiency, low dead weight and volume,
possibility for auxiliary impact (electric, magnetic, etc. [3]), and medium exchangeability would be also demanded for a number of applications.

Liquid media were considered because of easy desired contents preparation (properties variation) [4], well developed precise dosing (down to \(10^{-15}\) L and 0.1% stability [5]) and feeding systems. The main problems at laser impact on liquids are energy dissipation [6] and droplets formation due to splashing [7], leading to poor performance [8] even after application of special tricks like viscosity increase [9], thin films [10], cavities or droplets [11] formation. To the best of our knowledge, this trade-off has never been considered before [12] in terms of in situ solidification of a supplied liquid.

Solidification can be induced by several means, the fastest and the most temporally and spatially controllable ones are light and electron beam curing [13]. Curing time is about \(10^{-2}\) s and layer can be varied \(10^{-3}\) to \(10^{-7}\) m. At high laser power irradiation with certain wavelengths, curing may take place at impact itself, i.e. no pre-pulse is needed [14] since curing speed is proportional to light intensity square root. Similar to photocondensation of supersaturated vapor, solutions of numerous substances can undergo light-induced crystallization [15]. This process typically takes tens of seconds, limited not to the impact zone only, but also throughout the entire available volume eventually.

Medium supply, dosing and positioning are now well developed in paper (inkjet, laserjet, sublimation, UV) and 3D-printing (extrusion, light polymerization, powder bed, laminated, wire) [16]. However, supply system reliability is generally defined by number of moving parts and mechanisms complexity. For liquids, some physical effects could reduce need for moving parts: capillary effect, ultrasound (cavity resonance, capillary effect enhancement) [17], electro osmosis, electro spray [18], magneto hydrodynamic (MHD) [19], piezoelectric [20] and magnetoelastic [21] seem to be most applicable. For bulk solids dosing is not needed, and supply could be substituted by surface laser scanning system. Although electrooptical [22] and linear drives [23] need high voltage and current sources respectively, that is not always possible, e.g. due to electromagnetic compatibility issues.

The analysis of solid and liquid supply and dosing systems reveals that combination of their advantages and the partial elimination of disadvantages is possible in transition systems in which WM transportation, supply and dosing are performed in liquid state, but directly before impact increase of viscosity or solidification is induced due to physical or chemical processes. To conclude, among variety of solid and liquid supply systems developed there’s still no universal solution for laser plasma generators. Main shortcomings are related to exact medium and system performance restrictions or to laser plasma production poor performance. The aim of our research was to review state-of-art for fine dosing supply systems; to find out possible benefits of using special media and additives; to develop approaches to trade-offs resolution at working media supply systems choice.

2. Review of working media supply systems

2.1. General considerations

For solids SS, a number of advantages over liquid is often indicated in terms of ease of implementation, compactness and reliability, however, this is not always true. The former are not so good in WM utilization rate and dosing accuracy. Multiple irradiation of the same area often leads to GPF axis deviation from the normal to the target surface [24].

The development of microfluidics [5] allows to reduce WM supply discreet to \(~10^{-15}\) L (e.g., Eppendorf FemtoJet), which is unattainable for gases and allows significant increase in the utilization rate of solids. Droplet formation systems from inkjet printing, pharmacy (Lab-on-a-Chip), analytical chemistry (in particular, chromatography – http://dolomite-microfluidics.com) can be used also. There are commercially available solutions designed for melts microdosing at 250 °C (Microfab MJ-SF).

Droplets can be irradiated directly at SS output, or those can be deployed on an intermediate surface of single or multiple use. Direct evaporation limits the possibility of additional impact on the droplets. In addition, as a result of physical and chemical processes associated with direct evaporation, the nozzle may be clogged, so reliability and lifetime are reduced. Intermediate surface allows to get rid of these shortcomings, moreover, using several supply organs, WM flow and reliability of the
system as a whole could be improved. Intermediate surfaces (volumes) are also convenient for WM components mixing immediately before exposure. There are almost no data published on dosing stability affecting operation stability (0.1% vol. is stated for Fluent MFCS). Formation of a single dose by multiple smaller ones could resolve this issue to some extent. Laser ablation of a liquid on intermediate surface is investigated in [20]. Physical processes of droplet formation are discussed in details in [25].

To reduce parasitic droplets formation [4], it is necessary to use viscous [7], dilatant, electro- [26] or magnetorheological [27] fluids, as well as porous walls [28] or other methods of thin films formation at surface [29] and spatial limitation of WM volume [30] (including magnetic field [31]). However, repetition rate of highly viscous fluids discrete supply may be insufficient. Another advantage of liquid SS is possibility of easier plasma flow redirection (e.g., using gimbal). To increase the efficiency of energy conversion stored in a liquid, a scheme similar to that proposed in [32] for gunpowder can be used: the fuel charge is fed to the working chamber (similar to artillery systems), where its initiation occurs; the presence of an extended cylindrical section (barrel) ensures the completeness of the reaction and homogenization of its products.

2.2. Implementation examples for liquids

For exhausting chambers, maximum repetition rate is limited by piezoelectric membrane performance (~10³ Hz) and WM viscosity (Fig. 1a). By changing the voltage applied to the membrane or the opening time of the shut-off valves, WM mass flow rate can be adjusted according to the mode of laser exposure. Commercial example for such system is 0.5–5 μL/pulse microdosing device 7616 produced by Buerkert. Piezoelectric UV prinheads form droplets down to 4 pL (~20 μm dia.), some of those allow droplet volume adjusting in 6–42 pL range. In [24], the nozzle was formed in the membrane itself. A similar SS uses bimetallic or shape-memory material [33] plate instead of a piezoelectric membrane. Plate heating element used in that scheme restricts pulse repetition to 10–20 Hz due to thermal processes inertia. Significantly lower energy consumption at a comparable operation frequency can be provided by electrostatic pump with passive valves, its performance depends on the control voltage and is adjusted with 1 μL/min accuracy [34]. In principle, electrostatic drive has the lowest response time of ~100 μs. A similar system, combining transport and dosing function is possible with magnetorheological elastomer [21].

Worthington jet [35] is proposed for microdroplets (~30 μm) formation using relatively large (~2 mm) nozzle (Fig. 1b) in [36]. The active element is a membrane connected to an electromagnetic linear drive generating mechanical pulses of ~2 ms. A similar SS without microdroplets formation is considered in [37]. It is shown that for 2 mm dia. nozzle, surface recovery time due to surface tension forces is ~6 ms (τ=(ρR²/3σ)⁰.³). No need in micro-nozzle is the main advantage of this method, the latter significantly reduces SS reliability due to possibility of clogging. In [38], it is proposed to use laser-induced shock waves to generate pressure in similar case: high pressure pulse leads to liquid velocity of 300 m/s at the output, which results in high recoil momentum (this scheme was proposed for non-contact subcutaneous injection of drugs). Longer radiation pulses lead to increase of the mass flow rate and decrease of droplets speed; long sprays appearing due to this are demonstrated in [39]. Microsprays can be generated at flow through a slit same as through a round hole [40]. The pressure can also be created due to the pyroelectric effect [41].

The principle of thermoelectric capillary system is based on the formation of air bubbles at local heating and boiling of the liquid. A similar system is used in some inkjet printers, so it is well-developed and stable, the cycle can be repeated at a frequency of ~50 Hz. Heating can be also performed at laser irradiation of an absorbing capillary coating. Based on the scheme proposed in [42], adding a lobe valve, the inlet and outlet can be performed from the opposite sides. In modern printing systems, capillary is compressed with cylindrical piezoelectric element [20], which significantly increases repetition rate and reduces energy consumption. Droplet formation can also occur in special devices for the production of powders [43]. All three systems described above can supply from 10⁹ to 10⁶ L to the working chamber in one cycle, which makes them very attractive. The former two allow
it to be adjusted within a certain range. In addition to piezoelectric, thermal expansion, magnetostrictive and shape-memory effects can be used. Combination of spring and solenoid used to control the liquid flow regulation valve through a 25 or 50 µm dia. nozzle located in a pressure buffer chamber (dosing discreet ~3–4 µL in <1 ms) is proposed in [44]. Such system consumes ~0.1 J/pulse [45], leakage through the valve at a pressure drop of 10^4 Pa is ~0.1 µL/s. The patent [46] considers a number of such SS where flow rate of WM under pressure is regulated by shut-off valves. The piezo drive [47] in these systems is more preferable than solenoids, since the latter are not compact enough and consume more energy.

Another group of SS is based on pushing liquids through a porous wall [28] or capillary [48] (array of capillaries). Capillaries can be made of glass, quartz, ceramic or metal. Capillaries and porous walls have large friction losses, which imposes restrictions on WM maximum viscosity, makes to use relatively powerful pumps (pneumatic, mechanical piston or peristaltic) or create large pressure gradients in other ways. Capillary effect can be enhanced using ultrasound [17], electric osmosis (http://www.xrayoptic.ru/pump.htm) or electromagnet (for colloids containing ferromagnetic nanoparticles [49]) – all these means do not use any moving parts. In case of failure of such amplification circuits, system can remain partially operable. Ultrasonic capillary effect increases height and speed of the liquid column by an order of magnitude, which is important for high-repetition rates. It is shown in [50] that for effective use of surface acoustic waves in capillaries, the channel diameter should be >10µ. The permeability of membranes can also change when exposed to light, because this phenomenon is resonant, selective permeability can be induced. Ref. [28] is the only paper considering laser ablation of fluid transferred through a porous ceramic wall. Such system provided necessary mass flow rate; however, it can be very sensitive to clogging of the pores. Physical bases of fluid motion in micro-, nanocapillaries and porous media are considered in [51]. A similar system demanding less pressure gradient (10^1–10^3 Pa) is considered in [52] for laser drilled holes (~10 µm dia.) array in steel substrates – this method provides easier change of impact area as it is worn, and if necessary, making new or changing size of existing holes. The droplet size was found to be only slightly dependent on pressure gradient, which is convenient for adjusting the mass flow rate (fits linear). Droplets size dispersion was found to be relatively small, but it is not always possible [53], and often this problem is solved by surfactants. The electric spray is used in mass spectrometry and colloidal systems: a high positive potential is supplied to the liquid in the capillary, as a result of which the flow of drops rushes towards the collector (Fig. 1c). A liquid was fed into the capillary zone by rollers due to surface tension forces in [54]. A detailed overview of different micro pumps is given in [55]; physical processes of their operation can be found in [56].

Liquids can be encapsulated in a carrier tape [57], like bubbles on a packaging film; those can also impregnate porous solid matrices [58] (including aerogels [59]) or fill cavities at solid surfaces [24]. Droplets on the surface can be formed at areas with selective wetting [60] (e.g., electrically stimulated [61]). In these schemes, it is also possible to regenerate films or micro volumes of WM, including subsequently undergoing phase and chemical (polymerization) transformations both in the near-surface layer (for clogging) and throughout the volume (crystallization of gallium [61]). The simplest and most reliable system of liquid layer regeneration is self-recovery of ferrofluid [62] at magnetic substrate (or substrate over a magnet to reduce thermal loads on the latter – neodymium magnets can lose their properties when heated to 80 °C, their demagnetization is ~2%/year). Ferrofluids are known for their ability to move and form itself without applying mechanical force [63], just following the magnetic field that could be easily arranged with electromagnets, viscosity and virtual hydrophobicity could be also adjusted proportionally to the magnetic field strength and magnetic particles size [64] (electrorheological effect leads to viscosity increase by 5 orders of magnitude in milliseconds, but at 10 kV/cm electric field strength [26, 65]). The presence of axial magnetic field increases ferrofluid viscosity [66] in the impact zone and prevents the expansion of the droplet phase [67], but also increases GPF expansion rate [31] and collimation [68]. That would reduce mass loss and make plume more homogenous. Thin layer of ferrofluid forms conical droplets (~0.1–0.3 mm) around the magnetic field inhomogeneities, which can serve as a dosing system and prevents scattering of energy in the
volume of the liquid. The use of special magnetically oriented guide fibers [69] or capillaries allows to significantly expand the range of WM available. Nanolitre droplets can be formed when ferrofluids are drawn by magnetic field through the [19] membrane. The amount of stock materials making a liquid magnetorheological is of 10% vol. order.

![Diagram](image)

Figure 1. Supply systems for liquids: membrane-driven chamber (a), Worthington jet (b), and electrospray (c);
1 – liquid inlet; 2 – membrane (piezoelectric, bimetallic, shape memory, magnetoelectric); 3 – orifice options, 4 – outlet options; 5 – shock generation; 6 – droplet.

2.3. Implementation examples for solids
One of the main advantages of solids is ability to stay in direct contact with vacuum ($10^8$–$10^{12}$ Pa) for a long time without significant losses, while liquids will evaporate (except for some special ones). Although for some solid polymers, evaporation rate can reach 10% per year or more. Solids make less demand to ensure thermal regime of storage and transportation systems. Solid SS are more developed compared to liquid because of easier implementation. The simplest scheme of such a system is a screw-nut pair. The ratio between laser focusing spot and rod diameter should be selected for maximum WM utilization. It is difficult to fulfill this condition for systems with low-power lasers. This scheme has a number of limitations, associated primarily with the amount of WM stored and low coefficient of its utilization. Linear movement of WM is available down to $\sim$100 nm. An improved version of such SS is proposed in [70]: the cam profile provides movement by one laser focal spot per step; this allows the most complete use of WM.

It is possible to implement a replacement system for the whole disc or regeneration of the WM layer. The main disadvantage of such a system will be a low WM layer to substrate disc ratio if no regeneration is used. The advantage of this system is that it is well developed in computer technology, same as WM layer regeneration from a liquid state (spin-coating) matching CD and HDD spindle speeds. System with helical movement was used in [71]. For discs flat surface, it is necessary to use 2 motors (rotation and linear movement), which reduces reliability. For cylindrical surface, one motor and the screw–nut transmission are needed (the second motor can be used to reduce the axial step). In pulsed mode, the disc radius, and hence the amount of WM, is limited by the drive angular step. For big diameters, the linear step can be significantly larger than the characteristic size of the crater, which leads to WM utilization rate decrease.

The following group uses tapes, those can be considered in terms of laser source position and GPF direction: R-mode (Fig. 2a) and T-mode (Fig. 2b). For simplicity, we consider the case of a two-layer tape (WM and substrate), although the tape can be both single-layer (WM only) and three-layer (including confining layer). In such systems, it makes sense to use linear focusing rather than point to increase WM utilization rate [72]. At tape continuous movement, WM edge angle may change; since GPF axis is normal to the irradiated surface, this leads to its unpredictable deviation. To eliminate this defect, it is necessary to move the fuel strip in a discrete manner so that the untouched surface is irradiated. As an option, WM layer can deposited in isolated dashes [73]. In R-mode, GPF is formed at
the irradiated side of the tape. A significant disadvantage of this method is the possibility of contamination of transport optics with ablation products. Less significant drawback is that part of the radiation can be absorbed by the GPF, which on the one hand increases the effective evaporation threshold, and on the other – increases flow temperature, and hence its speed. In R-mode, it is possible to use single-layer tapes and metals (or other materials with high linear absorption coefficient). According to [72], R-mode is better for short (nanosecond) pulses irradiation. It has been shown for R-mode [9], that transparent confining layer (including initially liquid) above the WM can lead to significant (up to 5 times) increase in recoil thrust (determined by confining layer viscosity and thickness), however, the momentum coupling coefficient is reduced. Hexane, ethanol, and water were deposited on (CH₃O)₅ WM layer in [29], this led to a significant increase in the ablation efficiency. 3D printing technologies [73] can be used for 5–100 μm WM layer regeneration, as well as adsorption (e.g., electrostatic, magnetic) and desorption of wastes. It was also proposed [74] to use an intermediate layer of energetic material, to initiate processes leading to removal of the outer layer region matching the radiation focus spot. This fragment may subsequently be vaporized imparting strong recoil (fragment velocity ~1.5 km/s [74]).

Extrusion SS is based on pulling the filaments. Dead weight of the WM in this case tends to zero, and radial thermal conductivity losses at laser irradiation are reduced. WM flow can be easily regulated and controlled, the implementation of such a scheme is fundamentally very simple, and its implementation is well developed in FDM 3D-printers. Its disadvantages are in high probability of filament wrapping (if it is thin); high sensitivity to focusing accuracy and radiation irregularity. To eliminate the latter, it is necessary to build a complex optical system. In tape and extrusion systems, materials with shape memory (e.g., TN alloys (Nitinol)) can be used to improve reliability (due to no electric drives). The formation of filaments can occur from liquids (including melts – Fig. 2c). It is also reasonable to use piezo motors of various designs, because those provide minimum discreet of linear or angular displacement and have no restrictions for use in vacuum, unlike many electric motors.

A lot can be taken form 3D printing, by principle of WM layer formation those can be divided into the following groups: a selective treatment of the powder (3DP, SLS, DMLS, EBM, SHS) or solution (SLA, DLP, CLIP) in the bath; extrusion (FDM, DIW), external fusion of supplied filament (EBF), powder (DED) or the curing of the photopolymer (PolyJet, MJM); layer-by-layer cutting of the desired contour with the subsequent lamination (LOM). Technologies of the impact zone positioning: 3 axes Cartesian; parallelogram (Delta-robot) with print head tilt possible; self-propelled modules (Foundation robotic complex). Modern commercial FDM printers have 20 μm layer thickness resolution, PolyJet – 16 μm, DMLS – 20 μm, SLA – 25 μm, DLP – 50 μm, SLS – 60 μm. Lateral resolution of DLP technology has reached 100 μm, SLA and PolyJet – 150 μm, FDM – 300 μm, DMLS – 380 μm, SLS – 400 μm. The record accuracy among commercial products is known for wax printing with piezoelectric head (SCP): axial resolution reaches 6.25 μm, and lateral – 3 μm. For new CLIP technology the accuracy of 1–3 μm has been demonstrated. However, special laser lithography systems have sub-micron resolution in all axes (Nanoscribe). The latter, as well as PolyJet, CLIP, DLP, and SLA are limited to photopolymerizing materials. For FDM, thermoplastics, eutectics, metallic and modeling clays, etc. can be used; for DMLS almost any metals and alloys (powders) are suitable, and for SLS – even ceramic and polymer powders. Printing speed is strongly influenced by the technology used and accuracy, e.g., for FDM the order those vary from 40 to 1800 mm/s, and volume printing speed can reach 1 cm³/s or more.

The proven lifetime of commercial FDM printers is 160 thousand hours, or 3.2 m³ of thermoplastic. However, it should be noted that nozzle is considered as a consumable (its life is highly dependent on its manufacturing quality, materials used and filament purity; the order of its lifetime is 10–1000 hours, or 0.1–10 L). For industrial 3D printing, not cheap, but reliable nozzles are made of sapphire, those have a lifetime warranty. Also, 3D printing with several materials is being developed, which allows increasing reliability by duplicating with ‘hot’ replacement. Thus, problems of 3D printing reliability and solutions for many of those are well known nowadays (a matter of price).
2.4. Exotic options
An electro-optical system for 3D laser scanning of WM surface was proposed in [22]. Development of 3D printing significantly contributes to increasing the speed and reliability of such systems [75]. There are also alternative non-electrooptical scanning system: galvo (has limitations on the aperture of the beam ~14 mm), gimbal (consumes a relatively large amount of energy), Risley prism (not sensitive to vibrations). In this case, there is no SS at all; the design of WM block can be simplified geometrically, consist of several different layers in accordance with the technology process. Spot transfer speed (~10 m/s) similar to galvo-scanners can be achieved using Delta-robots [76], the latter has been tested under severe operating conditions. The advantage of using such a scheme with 4 degrees of freedom is the possibility of impact on almost any shape surface and at any angle. Existing precision dynamic focusing systems (Han's motor) provide some of these capabilities, too.

Supply in revolving blocks is considered in [24] (Fig. 2d). The peculiarity of this approach is that there is no need to focus laser radiation in a certain area of WM, because it explodes when sufficient energy is applied anywhere within the block. The set of blocks can follow the technology process. More exotic SS was proposed in [73] for loading of carbon balls of ~0.5 mm dia. The obtained values of the specific impulse ~7–18 s were very low due to the incomplete combustion of such large particles induced by 40–300 mJ laser pulses. Given that specific ablation rate usually does not exceed \( 10^4 \) g/J, balls should be at least 2 times smaller in diameter for efficient combustion. Microparticles down to 1 µm can be produced by UV curing of sprayed polymers with repetition rate of 100 Hz or more.

For powders, pneumatic and SS similar to some liquids can be used, some are well developed for stereolithography (3D-printers) [77]. It is reasonable to store and transport such WM loose, and to supply to the impact zone bonded [78]. Powders can be deposited on intermediate surfaces by magnetic or electrostatic forces (similar to laser printer), sintering (for example, laser), curing (drying) of liquid buffer. For porous WM, thermal and mechanical losses are minimized, and a large specific intensifies chemical reactions. Another SS was proposed for powders in [79]: laser-induced shock wave leads to metal foil high-speed deformation, powder dose from preliminary placed on its surface acquires a speed of ~600 m/s and crosses laser waist. Such an injection system can be implemented with liquids also, and ejection made more simple and reliable using piezoelectric drive, electric or magnetic field.

Pneumatic SS for nanoparticles was used in [80] to form tightly directed narrow stream by means of aerodynamic lens. Electrostatic injection of particles to form aerosol was proposed in [81]. Although, such SS are not suitable for pulsed operation, for high-frequency pulse-periodic or continuous mode it can be very useful.
3. Conclusions

Our brief review of condensed working media supply systems allows to conclude the following:
- there are no practical restrictions for individual parameters (single dose size, dosing stability, frequency of sampling, integral mass flow, lifetime);
- the number of supply systems matching several, often contradictory, requirements simultaneously is limited;
- conditions of SS use (vacuum, temperature, etc., impose additional restrictions, which arise the main problems to resolve.

General guidelines for choosing and adapting working medium supply system are as follows:
- transportation and dosing should be carried out in fluid-like state (aerosol, liquid or colloidal solution, loose substance), if this is not possible, then it might be reasonable to switch from supply system to laser scanning of WM surface;
- before exposure, WM should be applied to the intermediate surface (or to be confined in some volume), which reduces the risk of damage to the dosing system and is convenient in terms of multiple supply use of same or different media (components);
- the intermediate surface must be capable of re-use, monitoring its condition, timely replacement or cleaning if necessary;
- SS should provide the possibility of drives duplication in case of failure, avoid irreversible jamming or damage to the intermediate surface or blockage of the paths (piezoelectric effect, electric and magnetic fields are preferred).

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