



## Status and demand of research to bring laser generation of nanoparticles in liquids to maturity



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### ABSTRACT

Nanoparticles are already implemented as functional elements on surfaces and into volume, but also in hybrid nanostructures. Each application requires specific features regarding size, morphology, surface chemistry, purity, colloidal stability, defects, or doping. However, integration of the “nano-function” into products is still limited due to drawbacks of gas phase and chemical synthesis methods regarding particle aggregation and contamination by adsorbates causing deactivation of their surfaces. In addition, thermodynamically-controlled synthesis methods naturally face limited access to alloy nanoparticle systems with miscibility gaps. The development of new synthesis methods which can be reliably scaled up to industrial levels of production is mandatory to overcome these limitations and then widen the application perspectives of nanomaterials. Since the nineties, Laser Ablation in Liquids (LAL) has proven its reliability and its versatility to synthesize colloidal nanoparticles. More generally, laser/matter interaction in liquids offers several synthesis routes for nanoparticle generation. In addition to LAL, Laser Melting in Liquids (LML), Laser Fragmentation in Liquids (LFL), as well as pulsed Laser Photoreduction/oxidation in Liquids (LPL), offer different routes to obtain colloids with controlled nanoparticle sizes. We will present a digest of the breakthroughs achieved in the last years not only on the synthesis control, but also on the understanding of the basics. These achievements suggest that laser generation of nanoparticles in liquids is mature enough for industrial outlets of colloid production, with series products likely to approach the real world in the near future.

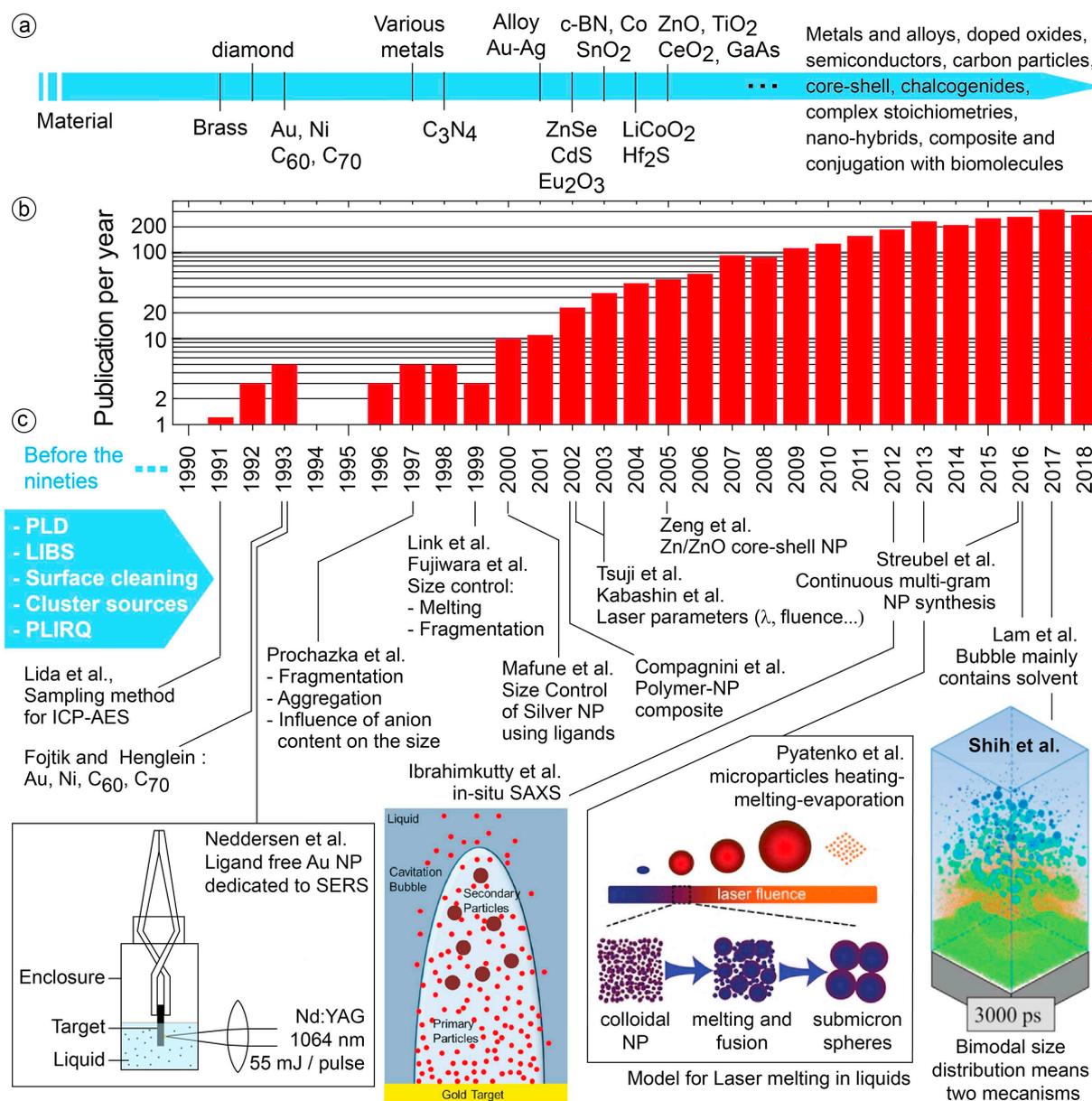
### 1. History

Since the nineties, laser ablation in liquids has proven its reliability and its versatility to synthesize nanoparticles, and reviews are available (see Fig. 1) [1,2,3,4,5,6,7,8,9,10]. The method appears with a set of works published in the early nineties. Fig. 1c shows a digest of the key findings in laser synthesis of colloids. In 1991, Lida et al. [17] were looking for a solid sampling technique to prepare samples for inductively coupled plasma atomic emission spectroscopy (ICP-AES). They described the production of “a suspension consisting of fine particles of around 1  $\mu\text{m}$  or less” which can be directly introduced into an ICP. A brass sheet was ablated with Nd:YAG laser, and the Cu:Zn ratio in the colloidal suspension appeared to be identical to the one of the target. The method was called LALM (Laser ablation in a Liquid Medium). However, the word *nanoparticle* was still not used. Between 1992 and 1993, several teams have reported the synthesis of carbon based materials [18,19,20,21], but the synthesis processes were not understood at that time [10]. Fojtik and Henglein [19] performed laser ablation of graphite microparticles suspended in toluene with a ruby laser and managed to produce carbon clusters, including C60 and C70. Ogale et al. [18] produced micro diamonds, from 5  $\mu\text{m}$  to 20  $\mu\text{m}$ , by laser irradiation of a graphite target immersed in benzene, using a pulsed ruby laser. In 1993, Neddersen et al. [16] were looking for surface-enhanced Raman spectroscopy active colloids. Stable colloids of Ag, Au, Pt, Pd and Cu nanoparticles were prepared by ablation of metal targets in water and organic solvents using an Nd:YAG laser. A mean size of 20 nm was reported for the Ag nanoparticles. The main advantages of the laser ablation in liquids were then explicitly described: “A new and

*highly promising method for preparing metal colloids is described that eliminates some of the problems associated with the chemical procedures [...] Advantages of this method include the simplicity of the procedure, its versatility with respect to metals or solvents, and the absence of chemical reagents or ions in the final preparation.”* The experimental conditions described in this pioneering work are still the most widely used, including the laser source, its focusing, the use of a batch chamber, and the target motion.

With the increasing number of works, it appears that laser ablation in liquids supports complex mechanisms which has to be controlled or avoided, such as the post-irradiation of the produced colloids. In 1997, Prochazka et al. [22] would like to synthesize size-controlled silver nanoparticles dedicated to SERS. In their contribution, they addressed three topics: the achievement of nanoparticle size-reduction by laser fragmentation of large particles, the aggregation and the sedimentation occurring with time in laser-generated colloids, and the influence of the anions content on the size distribution of laser-generated colloids. To control the particle sizes, they achieved a two-step process, first nanoparticles are produced by laser ablation of a solid target, then the solution is irradiated to induce the fragmentation of the larger particles (> 40 nm). The fragmentation process is now specifically developed to produce well defined nanoparticles from larger ones, including metals [23,24,25,26,27,28,29,30,31], oxides [32,33,34,35,36], semiconductors [37,38,39], and copper hexadecafluorophthalocyanine [40]. In the Prochazka's pioneering work, the effect of the anions content on the size distribution is also shown, however without a positive effect on the colloidal stability. The reason is that the anions concentration used was too high. Indeed, it has been shown later that

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**Fig. 1.** Key findings in laser synthesis of colloids: (a) materials produced using laser ablation in liquids. Comprehensive reviews are available [1,2,3,4,5,6,7,8,9,10]. (b) Publication count from Web of Science™ (Thomson Reuters). (c) A digest of significant milestones (see plain text for details). Regarding the earlier experimental setups dealing with laser matter interaction, PLD, LIBS and PLIRQ stand respectively for pulsed laser deposition, laser-induced breakdown spectroscopy, and pulsed-laser-induced reactive quenching [11,12]. ICP-AES stands for coupled plasma atomic emission spectroscopy. The illustration come from Ibrahimkuty et al. [13], Pyatenko et al. [14], and Shih et al. [15]. The scheme of the original experimental setup from Neddersen et al. [16] has been redrawn.

the colloidal stability of metallic particles can be enhanced when the concentration of chaotropic anions in the solution is between a few tens and a few hundreds of  $\mu\text{mol}$  [41,42]. This method yields monodisperse particles, where the anion's action takes place already within the cavitation bubble on microsecond time scale [43].

In 1998, Wang et al. performed the laser ablation of a carbon target in ammonia, and reported the synthesis of Carbon Nitride C<sub>3</sub>N<sub>4</sub> [44,45]. It then appeared that atoms from the solvent can significantly contribute to the stoichiometry of the produced nanoparticles. Wang et al. named the method Pulsed Laser Induced Liquid-solid Interfacial Reaction (PLIIR) in accordance with the physico-chemical mechanisms they assumed [45]: "(1) The interaction of the laser beam with the bulk target [...] results in the formation of a high-temperature, high-pressure, and high-density plasma of evaporated materials. (2) The interaction of the plasma with the liquid at the interface. The high-temperature of the plasma can lead to the production and emission of a high level of positive ions and electrons

from the liquid interface. (3) Most of the positive ions from the solid target and liquid, respectively, react with each other at the interface between the plasma and liquid to form the nuclei of the nanocrystals [...] Therefore, with the plasma rapidly quenching in the liquid, the metastable-phase nuclei grow large. As the growth times of the nuclei are very short, all metastable phases formed in the reactive process may co-exist, after plasma quenching." Even if physico-chemical mechanisms are still under debate, some recent evidence from experiments appears consistent with some assumptions made in this pioneering work. Professor Sakka's group showed that in the early stage, the nascent plasma and the liquid partially merge [46]. They also showed the transfer of Li<sup>+</sup> and Na<sup>+</sup> ions dissolved in the liquid into the plasma using plasma spectroscopy [47]. The fast quenching of the plasma has also been documented. The cooling rate observed in laser ablation in liquids can reach  $10^{10}$  K/s at the early 200 ns [10], faster than the cooling observed in other methods [48]. As emphasized by Wang et al., laser ablation in liquids appears particularly

relevant to prepare metastable phase compounds, including diamond [10], cubic-BN [49,50], tetragonal nanophase of Ge [51], monoclinic  $\text{Gd}_2\text{O}_3$  [52,53,54], but also bimetallic nanoparticles frozen in metastable morphologies [55,56]. Following this pioneering work, several works report the contribution of the solvent in final products, even if the growth mechanisms involving solvent remain scarcely described. Indeed, carbon nitrides were observed after carbon ablation in ammonia [57,58]. Numerous oxides were obtained from laser ablation of pure metal targets in water [59,60,61,62,63,64,65]. Boron carbide  $\text{B}_4\text{C}$  particles were obtained after irradiation of boron powder in various organic solvents [66]. TiN and TiC were obtained from laser ablation of titanium target in liquid nitrogen [67] and dichloroethane [68], respectively. Large area  $\text{Cu}_2\text{S}$  nanorod arrays in the high temperature phase ( $\alpha$ ) are obtained by laser ablation of a copper foil in carbon disulfide [69]. Ablation of metals in organic solvents can also lead to core-shell structure with a shell consisting in carbon layers [70,3,71]. Based on a series of LAL and LFL works in acetonitrile, Choi's group systematically analyzed the carbon shell thickness into detail, and found a thicker shell with increasing nanoparticle diameter [72]. Interestingly, with high relevance for catalysis, these layers are nitrogen-doped graphitic layers on nickel nanoparticles, as the smartly chosen solvent was acting as both the carbon and nitrogen source. Marzun et al. recently identified the role of dissolved molecular oxygen on the oxidation of copper nanoparticles [73]. They also observed a carbon shell around copper nanoparticles when produced in acetone, attributing its formation to the well-known copper-catalysed C–C coupling. These carbon shells seem not to be fully tight, so potentially allowing molecular access to the copper surface (for catalysis), and at the same time providing oxidation protection.

In 1999, laser-induced melting is reported on gold colloids by two groups [23,24]. The laser-induced melting and evaporation of the nanoparticles, initially considered as a problematic side-effects during LAL, has gained a renewed interest in the last decade. Laser melting in liquids (LML) has been extensively studied [26,27,14,74] to reshape colloids [75,76,77] or to produce sub-micrometer spheres of high purity [78,79,80]. Koshizaki's group recently showed that sub-micrometer spheres created by LML bear up to 40% of the ideal tensile fracture strength calculated using density functional theory [81]. Hence, these spherical particles obtained using LML exhibit quite strong and unique mechanical properties, highly relevant for tribology. The same group has also elaborated a low-threshold random laser from a film composed of highly pure sub-micrometer-sized ZnO spherical particles [78,79].

In 2000, Mafune et al. showed that the addition of ligands in the aqueous solution helps to achieve a better control of the colloidal stability, as well as a size control of the produced nanoparticles [82]. Such a positive effect of the ligands has been observed for both LAL and LFL. It also appears that the ligands can help to prevent the formation of metal hydroxyde, as observed for ZnO synthesis [83,84], but can also lead to a control of the shape of the nanoparticles, as observed for  $\text{Ag}_2\text{O}$  [85]. One can find a comprehensive review on the use of ligands in Zhang et al. [6].

In the early days of the millennium, the basics of nanoparticle generation and excitation by lasers in liquids were laid, including Laser Ablation in Liquids (LAL), laser fragmentation in liquids (LFL), and laser melting in liquids (LML). Laser parameters have been addressed [86,87]. Synthesized materials are becoming more and more diversified (see Fig. 1a), and the number of contributions to the field is rising together with the number of contributors (see Fig. 1b).

More recently laser generation of hydrogen peroxide has been demonstrated [88], opening the door to photochemistry [89]. It leads to another strategy, which can be called laser photo-reaction in liquid (LPL), bringing closer laser generation of nanoparticles in liquids to photochemistry. Laser-mediated reduction of metal salts is developed to produce core-shell particles [90] or nanoparticles composed of materials with low miscibility [91]. Fluorine-Doped Carbon Nanoparticles are laser-generated from Hexafluorobenzene [92] and Bare Iron

Nanoparticles are laser-generated from Ferrocene Hexane [93].

Pulsed laser ablation in liquids is also used to produce precursor solution, and then a post-treatment is applied to the freshly produced colloid to control the crystal structure or the particle shape. Silver nanoplates are produced by addition of hydrogen peroxide in LAL-generated colloidal silver [94]. Pt/ $\text{SnO}_2$  nanocomposites are produced after ageing LAL-generated non-stoichiometric  $\text{SnO}_x$  nanoparticles in a solution of  $\text{Na}_2\text{PtCl}_4$  [95]. Laser ablation of a tungsten target followed by an ageing treatment [95,96] or an hydrothermal treatment [97] leads to various shaped particles of tungsten oxide  $\text{WO}_3$ . Ageing can also lead to phase transition, from LAL-produced amorphous nanoparticles into nanoparticles with well-defined crystal structure. Amorphous selenium nanoparticles evolve into trigonal Se [96]. Amorphous germanium nanoparticles evolve into a metastable tetragonal structure, and then transformed into the stable cubic structure [98].

Laser-based methods of generation of colloids are becoming more and more diversified with the aim to reach a better control of the product (size, morphology, crystal structure), which remains an inherent shortcoming of laser ablation in liquids involving highly transient physico-chemical processes. That is why many efforts are made to achieve a comprehensive description of these processes.

## 2. Understanding of the processes

Along all these years, many teams have sought to understand and describe the physico-chemical processes involved in the laser generation of nanoparticles in liquids. Many efforts have been made to develop *in situ* characterization. Fig. 2 outlines the different characterizations reported in the literature, as well as the provided information, and the reachable time scale for each experimental method. From these measurements some findings are now widely accepted. First of all, *in situ* time-resolved small-angle X-ray scattering [13,118,119,43], as well as light scattering experiments [102,101], not only showed that the nanoparticles are confined inside the vapor bubble, but also showed that nanoparticles are present from the early stage (see insets in Fig. 1c). In particular, light scattering experiments suggest that nanoparticles are present after a few hundreds of nanoseconds. This is consistent with the fast cooling of the plasma reported from plasma spectroscopy [10], keeping in mind that the fast cooling of a laser-generated hot gas or plasma leads to nucleation and growth of particles [131,48,132]. This scenario is also supported by numerical simulations. In particular, a breakthrough in the understanding has recently been achieved thanks to molecular dynamics. The Zhigilev's group has developed an atomistic simulation of the laser ablation of metal targets in liquid environment [15,133,134]. They clearly showed two mechanisms of nanoparticles generation occurring in less than a few nanoseconds for femtosecond and picosecond pulses: (i) a rapid nucleation and growth of sub-10 nm particles in the water-metal mixing region, and (ii) an instability of the superheated metal layer formed at the interface with water leading to particles of a few tens of nanometers. They then succeeded to explain the bi-modal size distribution frequently reported from transmission electron microscopy [135,136], but also from SAXS measurements: a first population of nanoparticles of around 10 nm diameter, and a second population of larger nanoparticles with diameters of a few tens of nanometers.

Nevertheless questions remain. The process leading to the fast vaporization of the solvent [116], to its decomposition, and then to its reactivity [72] is not fully described. The transfer of solvated ions ( $\text{Na}^+$ ,  $\text{Li}^+$ ) into the plasma has been reported [47], which is unexpected for standard vaporization. It has then been suggested that for nanosecond pulses the water experiences explosive boiling. This echoes the questions on the size quenching induced by anions for metallic particles, or ligands in general. The size quenching induced by the chaotropic anion's action during laser synthesis of metallic particles in a solution of low salinity happens already in the vapor phase of the cavitation bubble [43]. Drastic size quenching observed with ligands [130,111,84] is also

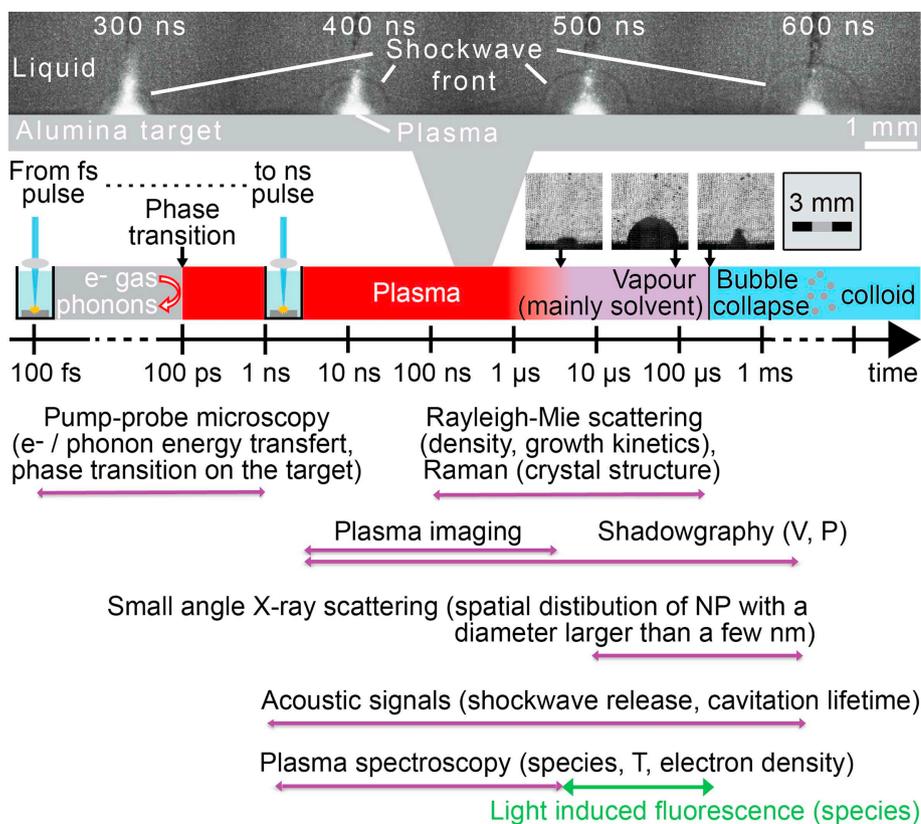


Fig. 2. Overlapping time scales of laser synthesis of colloids. At the top, the spatial distribution of the plasma is observed from shadowgraph imaging, as well as the laser induce shockwave [10]. Three pictures from shadowgraph imaging show the cavitation bubble (ablation of a carbon target, 5 ns pulse duration, 45 mJ/pulse, 355 nm) [10]. The time frame gives some insight into the different characteristic times of the main physical process in laser ablation in liquids: energy transfer from the laser excited electron gas to the matrix (a few ps), phase transition of the target (above 100 ps), plasma lifetime (a few  $\mu$ s), and bubble lifetime (a few hundreds of microseconds). *In situ* characterization has been developed to obtain information on the processes involved during laser ablation in liquids: Pump-probe microscopy [99,100] Rayleigh-Mie scattering [101,102,103], Raman scattering [104], plasma imaging [46,105,106,107,108], shadowgraph imaging [103,109,110,111,112,113,114,115,116,117], small angle X-ray scattering [13,43,118,119], acoustic signals [111,120,121], and plasma spectroscopy

[10,47,101,107,112,122,123,124,125,126,127,128,129,130]. The arrows display the reachable time scale for each experimental method, taking into account the integration time of the signal. The main information provide by each experimental method are also displayed. Light-induced fluorescence has not been reported yet in the framework of LAL, but it would be the next step to obtain deeper information on the intermediate species.

consistent with an early size quenching during the cooling of the plasma, and then to an early penetration of the ligands into the system. On the other hand, it is difficult to imagine that the ligands were not decomposed when they interact with the plasma, since the decomposition of the solvent can occur [92,93]. As a matter of fact, hydrogen and oxygen emission has been reported during laser ablation in water [137], as well as during laser irradiation of colloids [137,138,139,88].

At last, if one accepts that the nanoparticles appear early in the plasma, the maturation of the particles during whole lifetime of the vapor bubble remains poorly addressed. One can imaging Ostwald ripening, coalescence, or aggregation.

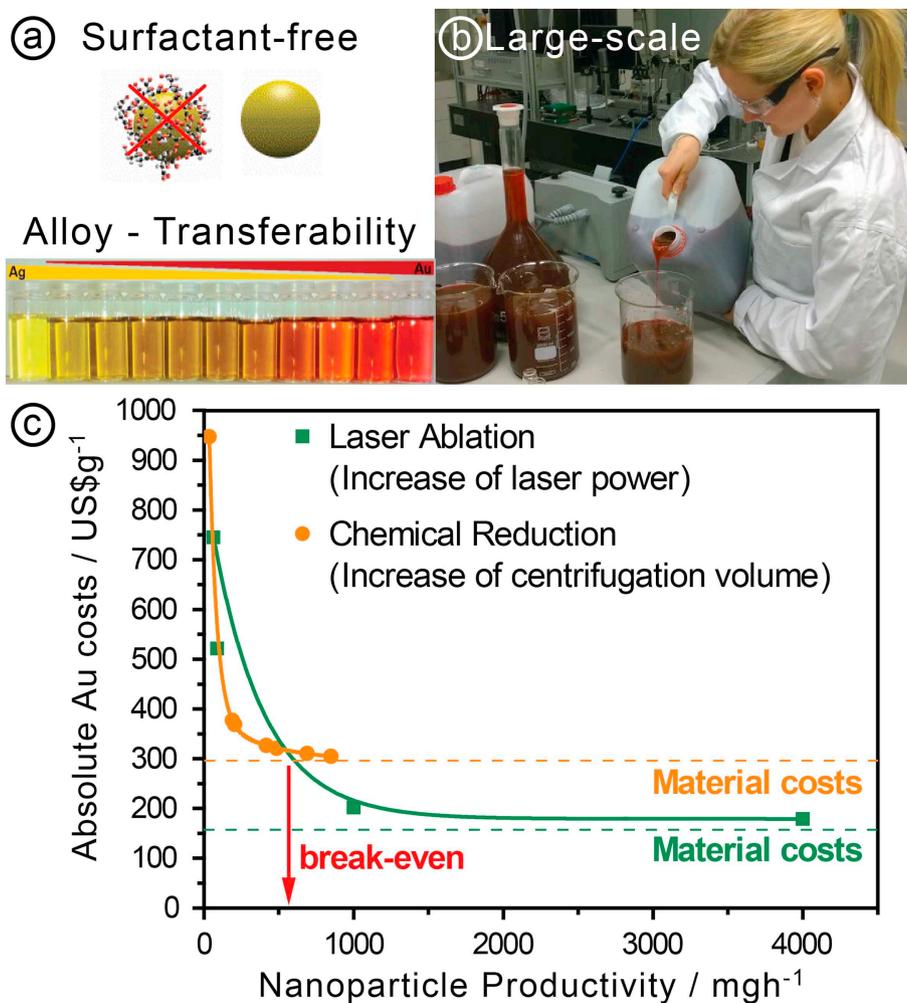
### 3. Economical viability

Nanoparticles are widely implemented with a wide spectrum of applications such as optics, optoelectronics, electronics, biomedicine, pharma and health, catalysis, energy science, automotive industry, or nutrition. These high-end-applications of colloidal nanoparticles require very well defined material properties not being compromised by impurities, and downstream processability of colloids. Both criteria perfectly met by laser-synthesized colloids. In contrast to chemical routes, LAL does not require using surfactants or chemical precursors. Moreover, LAL can be performed in a wide variety of liquids beyond the standard solvents, including ionic liquids [142,143], and liquefied gas [67,144,145,146,147,148]. Hence, as an alternative synthesis route, laser generation of nanoparticles in liquids has proven its capability to generate ligand-free colloidal nanoparticles with high purity for a variety of materials [6]. Ligand-free surfaces are interesting for catalysis as it drastically increases the yield of particle deposition on the solid co-catalyst (that is used, e.g. for wash-coats or fuel cell electrode impregnation). This year, the first real fuel cell device has been demonstrated based on laser-generated Pt/C nanoparticles [149].

In materials science, alloy nanoparticles bear great potential [150,151,152]. Alloys illustrate one of the advantages of LAL with respect to usual chemical synthesis. The transferability of the laser synthesis route to a variety of materials further enabled high-throughput screening of molar fraction series, sometimes not accessible to usual chemical synthesis. Alloy nanoparticles series were synthesized (i.e. AgAu, NiMo, AuFe, AgNi, FeNi, AlMg) [153,140,154,155,156,157,55,137]. Interestingly, on the one hand, phase diagram seems to play a role in ruling the nanoparticles crystal structure and phase segregation, but at the same time, unusual structures difficult to access by conventional synthesis methods are yielded, indicating kinetic control.

Beyond the above examples, the versatility of the aforementioned synthesis methods involves a high potential for applications, including bioimaging [158,159,160], cancer therapy [161,162], antibacterial coatings [163], heterogeneous catalysis [164,157,95,8,165], photoelectrocatalysis [166], proton exchange membrane fuel cell [149], oxygen evolution reaction [36,167], hydrogen evolution reaction [168], water oxidation catalysts [169], plasmonic [170], Fano resonance [171], random lasers [78,79], photovoltaic [172,173,174], additive manufacturing [175,176], and nanoparticle-polymer composites [7,177,178]. Surface-functionalized nanoparticles can be prepared in a one-step process [158]. Looking at the application in life sciences, the biodistribution issue has been addressed [179,180].

Recently the Gökce's group achieved a crucial breakthrough for industrial outlets, but also for fundamental studies where a large amount of material is needed to conduct experiments and to address reproducibility, specifically for application in life-sciences. Good reproducibility and significant up-scaling of nanoparticle generation were achieved by a continuous flow synthesis using a high-power ultrafast laser system leading to a productivity of up to multi-gram, multi-liter colloidal nanoparticles [181,182]. LAL is then scalable and



**Fig. 3.** a) Laser ablation in liquids enables the production of surfactant-free alloys [140]. b) Dr. Galina Marzun handling multi-liter of laser-made concentrated colloidal gold (multi-gram). c) Absolute manufacturing- and labor costs for the synthesis of 1 g colloidal gold in dependence of the nanoparticle productivity for pulsed laser ablation of gold in water and chemical reduction.

(Figure from Jendrzej et al. [141].)

economically feasible [141] (Fig. 3).

Thanks to these advantages, companies are doing business since almost a decade with laser-generated nanoparticles, as well as LAL-made heterogeneous catalyst powders: Particular GmbH [183], the distributor Strem Chemicals, Inc. (USA), and the i-Colloid in-house branch of IMRA (USA) [184]. The materials produced support a concentration of at least 100 mg L<sup>-1</sup>. Biocompatible colloids and ligands-free colloids are available.

#### 4. Future challenge

The progress in these fields is biannually presented at the international ANGEL meeting [185], and special issues are published concomitantly [186,187,188,189,190,191]. It appears that the question of the nanoparticle formation is still only partially answered. Molecular dynamics as well as experimental developments tend to show that the processes occurring at the early time scales, ranging from several femtoseconds to several microseconds, define the characteristics of the produced nanoparticles [192]. Numerical and experimental developments are then mandatory to catch the early time scales. One of the main challenge for molecular dynamics simulation is to include laser-matter interaction for nanosecond pulses. Indeed numerical simulation assuming femto- and picosecond pulse duration cannot catch the whole complexity of the processes resulting from the plasma-laser interaction

which occurs for nanosecond pulse duration. Plasma/liquid interaction has to be addressed to understand the fast vaporization of the solvent, as well as the parameter favouring its decomposition and its reactivity. Light-induced fluorescence would help to obtain deeper information on the intermediate species.

The purity of the product, the selectivity in the crystal structure of the oxide for instance, a fine control of the particle size distribution, and a tight control of the morphology of the particles are shortcomings of the former laser ablation in liquids. As a consequence, the community has developed various strategies to obtain a tight control of the products, leading to various laser-based methods of colloids generation. However, efforts must be maintained to reach the tight control achieved by the chemical routes.

Last but not least, the aforementioned companies sell laser-generated metal and metal alloy colloids in the fine chemicals market. But there is a trend towards coming closer to application, e.g. colloids developed for biotechnology (lateral flow assays, IMRA), or so-called supported nanoparticles for heterogeneous catalysis in larger scale (catalyst powders, STREM Chemicals, Particular GmbH). On the one hand, this points out the maturity of the laser-based synthesis of colloids. But on the other hand, end-products based on laser-made colloids did not reach the market, yet. An industrial series product or manufacturing process based on laser-synthesized particles is yet to come.

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