

Article

Femtosecond Laser-Induced Copper Oxide Nanospheres on Copper Foam Surfaces

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Abstract

A facile and scalable strategy is presented in this work for the direct fabrication of binder-free copper (Cu) oxide nanospheres on the Cu foam surface via femtosecond (fs) laser ablation for energy storage applications, primarily in supercapacitors. XRD and EDX analyses confirmed the presence of Cu oxides. At the same time, SEM images indicated that the resulting Cu oxide nanospheres range from ~70 to 700 nm in size, with hierarchical surface features such as laser-induced periodic surface structures (LIPSS), which provide additional active sites for reversible redox reactions. The prepared fs laser-ablated Cu foam samples, with Cu oxide nanospheres (Femto-Cu), can store 8 to 10 times more energy than the bare Cu foam, with ~87.7% capacitance retention after 10,000 charging–discharging cycles. Further, in-depth kinetic investigations revealed that the charge is stored through both surface-controlled capacitive behavior and a diffusion-controlled mechanism. These findings highlight the effectiveness of fs laser-induced structuring in improving the charge-storage characteristics of Cu foam and provide a promising route for developing high-performance, binder-free electrodes in a single step.

Keywords: femtosecond laser; copper oxide nanospheres; binder-free electrodes; supercapacitors; electrochemical analysis

1. Introduction

The steady growth of portable electronics, electric vehicles, and renewable energy technologies has drawn significant attention to energy systems capable of delivering both high power and long-term operational capability [1–4]. Amongst the available energy storage systems, supercapacitors have attracted significant interest due to their ability to bridge the performance gap between conventional dielectric capacitors and batteries, offering fast charge–discharge ability, high power density, and high cycling stability [5–7]. However, their relatively low energy density remains a key limitation, making the design of next-generation materials a prime research focus [8–10].

Numerous materials like carbonaceous [11], transition metal oxide (TMO) [12], perovskite oxides [13], MXenes [14], conducting polymers [15], metal–organic frameworks [16], porous organic polymers [17], etc., are utilized as potential electrode materials for supercapacitor application, and among these materials, TMOs have arisen as promising pseudocapacitive materials because of their fast and reversible Faradaic redox reactions to store charge [18–23]. Due to the low cost, natural abundance, environmental friendliness, and high theoretical capacitance, the copper oxides (CuO, Cu₂O) stand out among TMOs [24–28]. Multiple studies have demonstrated that tailoring the nanostructures of



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Cu oxides, such as nanowires [29], nanoparticles [30], flowers [31], nanoribbons [32], and porous architectures [33], due to the large active surface area, can boost electrochemical performance and accelerate the diffusion of electrolyte ions [34–37].

Conventional synthesis techniques, including hydrothermal growth [38], chemical oxidation [39], and electrochemical anodization [40], face significant challenges, such as poor structural uniformity, limited reproducibility, low electrical conductivity, and weak adhesion between active electrode materials and current collectors [40–44]. In many cases, binders such as PVDF [45] and Nafion [46] are used to ensure proper bonding among active electrode materials and the current collectors in energy storage devices [47,48]. However, these binders often reduce the overall conductivity of the active electrode materials, so conductive materials such as carbon black are added to counter this effect [49–51]. To overcome the drawbacks of binder usage, a different strategy is needed to enable the easy production of binder-free, active-material-coated electrodes for energy storage systems. For this purpose, ultra-fast laser technology, such as femtosecond (fs) laser processing, can be used [52–57]. It offers a unique and versatile platform, as it can precisely modify surface morphology and chemistry without introducing thermal damage and, more importantly, can easily be reproduced [58–64]. This technology can also be used to produce binder-free coated electrodes with active materials on metal foams (Cu, Ni, etc.), especially oxides. The ultra-short pulse duration and extremely high peak power of fs lasers enable the generation of dense micro- and nanostructures and surface defects, which act as preferential nucleation sites for subsequent oxidation processes [65–70].

There are a few studies on the binder-free coating of active materials onto Cu foams, and one prominent study was undertaken by Wang et al. [71]. In this study, they fabricated 3D CuO nanoflower structures on Cu foam via fs laser-assisted anodization for supercapacitor electrodes and noted that the flower-like morphology provides additional surface area for more electrochemical reactions, thereby improving the electrodes' overall energy storage capacity. The prepared electrodes exhibited a high areal specific capacitance (C_{sp}) of $\sim 3348.6 \text{ mF/cm}^2$ at a current density of 1 mA/cm^2 within a 0–0.4 V potential window. Electrodes showed $\sim 82.5\%$ rate capability after 1400 charging–discharging cycles. However, the working potential window in this study is narrow and requires further study to broaden it. In another study related to the binder-free coating of polypyrrole (PPy) on Cu foam via fs laser, Maqsood et al. [72] demonstrated a significant improvement in C_{sp} over the bare Cu foam. Prepared fs laser-assisted PPy-coated Cu foam electrodes showed a C_{sp} of 148.5 mF/cm^2 at a 0.5 mA/cm^2 current density with a potential window in the range of 0–0.6 V. The electrode also showed strong durability, maintaining $\sim 78.6\%$ capacitance after 10,000 GCD cycles. This study was the first of its kind in which conducting polymers were coated onto Cu foam using an fs laser.

The present study reports a simple, single-step, and scalable approach to fabricating fs-laser-induced Cu oxide nanospheres directly on Cu foam, yielding a binder-free electrode for supercapacitors. XRD, SEM, EDX mapping, and electrochemical characterization were used to study the prepared electrodes in detail. Femto-Cu has Cu oxide nanospheres ~ 70 to 700 nm in size and delivers 8 to 10 times higher charge-storage capability than bare Cu foam, making it a potential electrode for energy storage systems.

2. Experimental

2.1. Materials

The 1 mm-thick copper (Cu) foam with over 99% purity and analytical-grade potassium hydroxide (KOH) were purchased from Tmax Battery Equipment Ltd. (Xianmen City, China) and Sigma-Aldrich (Burlington, MA, USA), respectively.

2.2. Electrodes Preparation

Binder-free Cu oxide nanospheres-coated Cu foams are produced through fs laser ablation, following previous work [72–74]. The laser processing conditions were adjusted by applying a 2W power, 20 mm/s scanning speed, 20 fs pulse duration, and 0.04 mm inter-spot spacing, and running the process as shown in Figure 1. The prepared binder-free Cu oxide nanospheres coated on Cu foam through fs laser ablation in this study will be referred to as Femto-Cu.

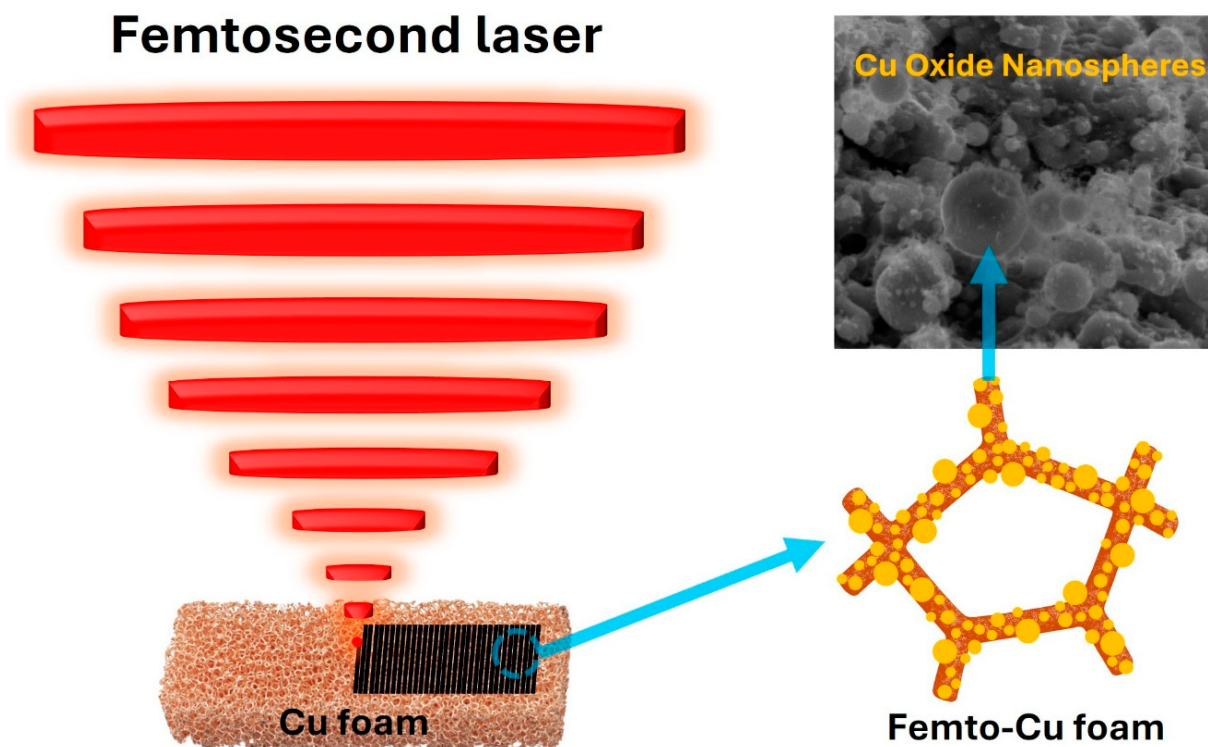


Figure 1. Schematic of femtosecond laser-induced Cu oxide nanospheres on Cu foam.

2.3. Characterization

XRD from Panalytical (Almelo, The Netherlands), SEM from TESCAN VEGA 3 LMU (Brno, Czech Republic), and EDS detector from Oxford Instruments (Bristol, UK) were used to acquire XRD spectra, SEM images, and elemental mapping. For electrochemical characterization, an electrochemical workstation from CH Instruments Inc. (Bee Cave, TX, USA) was used. More detailed information on the characterization tools and electrochemical equations used in this study is given in the Supplementary Data File.

3. Results and Discussion

Changes in crystal structure, composition, and phase are determined using XRD analysis. Figure 2a shows the XRD spectrum of both samples (bare Cu and Femto-Cu foams) with characteristic diffraction peaks around 43.32° , 50.49° , and 74.14° , consistent to the (111), (200), and (220) planes of Cu and well matched with the reference pattern COD# 96-901-2955, confirming that the crystal structure is preserved even after fs laser processing. Notably, the insert XRD patterns in Figure 2a for the Femto-Cu sample illustrate a weak and broadened peak centered around 36.49° , which is mainly attributed to the (111) plane of Cu_2O , indicating the formation of a thin, nanocrystalline oxide layer. However, other minor peaks around 35.67° could also be observed, characteristic of the (-111) plane of CuO thin layers; still, CuO_2 is predominant reactively sputtered [75–77].

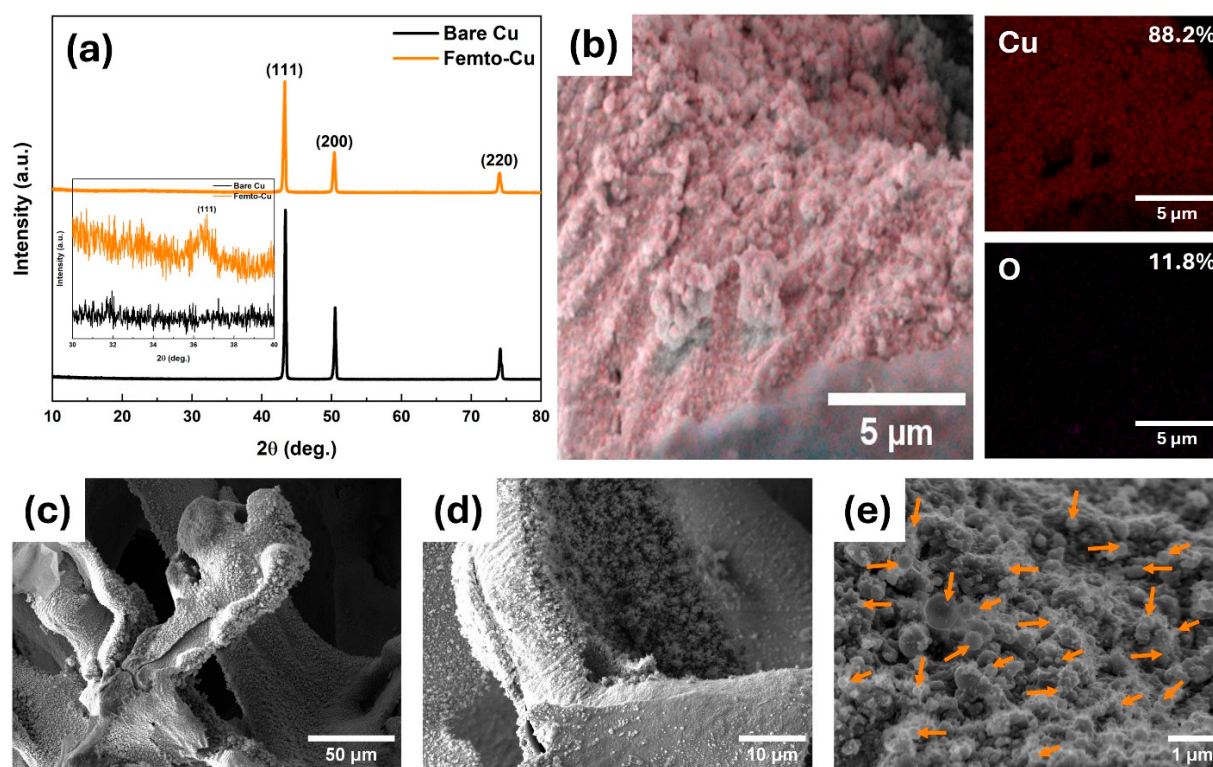


Figure 2. (a) XRD spectra of bare and Femto-Cu foams. (b) EDX elemental mapping of Femto-Cu. (c–e) SEM images of Femto-Cu foam (arrows point to the Cu oxide nanospheres).

Further, the presence and uniform distribution of Cu oxides on the surface of the Cu foam after fs laser ablation are confirmed through EDX elemental mapping and presented in Figure 2b, where the presence of Cu (~88.2%) and oxygen (~11.2%) indicates successful surface oxidation, while the uniform distribution of oxygen supports the formation of a continuous oxide layer across the fs laser-ablated surface. The morphological modifications induced by the fs laser irradiation are revealed using SEM images in Figure 2c–e. The microporous Cu foam framework is retained, as seen in Figure 2c, and the surface becomes rough and dense due to LIPSS, as illustrated in Figure 2d. Uniformly distributed Cu oxide nanospheres (~70 to 700 nm in size) can be clearly observed and are indicated by arrows in the higher-magnification SEM image (see Figure 2e).

These Cu oxide nanospheres were produced by fs laser-induced melting, ablation, and plasma-mediated redeposition [78–80] on Cu foam surfaces. Herein, during ablation, molten Cu rapidly cools and condenses within the expanding plasma plume, thereby nucleating spherical nanostructures. This morphology is thermodynamically favored due to its minimum surface energy and lowest surface-to-volume ratio [81–83], while the ultrafast quenching conditions suppress anisotropic growth, resulting in highly stable nanospheres, whereas in parallel, the oxidation of these spherical nanostructures in open air happened during and after plume expansion, causing the production of Cu oxide (CuO, Cu₂O) nanospheres at the Cu foam surface, and this repeated interaction of the fs laser pulse enhances this hierarchical and binder-free coating of Cu oxide nanospheres on Cu foam surfaces. A clear schematic of the process for producing fs laser-induced Cu oxide nanospheres on Cu foam is presented in Figure 1.

Electrochemical characterization is performed using a 3M KOH electrolyte solution and a 3-electrode assembly. The CV curves of the Femto-Cu electrode recorded at different scan rates (2 to 100 mV/s) in the range of 0 to 0.6 V are shown in Figure 4a. These quasi-rectangular CV curves with redox humps indicate that the charging behavior arises from the

combination of pseudocapacitive and EDLC behaviors but is dominated by Faradaic redox reactions [84,85]. Possible reversible redox reactions occurring from the Cu oxides and Cu foam surface are illustrated in Figure 3 [71,75], whereas the C_{sp} values calculated using Equation (S1) are presented in Table 1. The calculated C_{sp} decreases from 204.4 mF/cm² at 2 mV/s to 98.8 mF/cm² at 100 mV/s, typically due to partial ion diffusion at higher scan rates, as ions have less time to move and react. The CV curves of bare and Femto-Cu foam at 2 mV/s are shown in Figure 4b, where the significantly larger enclosed area under the CV curve and higher current response confirm the superior capacitive behavior of the Femto-Cu electrode. Quantitatively, the C_{sp} of Femto-Cu (204.4 mF/cm²) is $\sim 8\times$ greater than the bare Cu (25.8 mF/cm²), demonstrating the strong effect of the fs laser-induced nanospheres of Cu oxides formation at Cu foam surfaces.

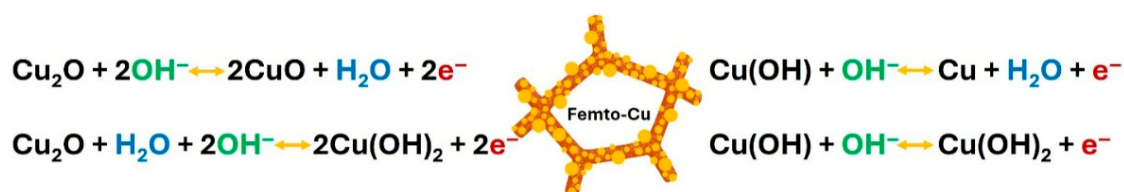


Figure 3. Possible redox reactions.

Table 1. Calculated C_{sp} .

From CV		From GCD	
Scan Rate (mV/s)	C_{sp} (mF/cm ²)	Current Density (mA/cm ²)	C_{sp} (mF/cm ²)
2	204.4	1	243.8
4	182.6	3	215.3
6	170.6	5	203.3
8	160.1	8	192.7
10	150.7	10	186.6
25	133.1	15	175.0
50	117.9	20	166.6
100	98.8	30	150.0

In Figure 4c, the GCD curves at various current densities (1–30 mA/cm²) for Femto-Cu show a nearly symmetric charge–discharge profile, indicating good reversibility, and as expected, the discharge time decreases with increasing current density. The C_{sp} values calculated using equation (Equation (S2)) are listed in Table 1, and these values decrease from 243.8 mF/cm² at 1 mA/cm² to 150.0 mF/cm² at 30 mA/cm², reflecting good rate capability even at higher current densities. To better understand the charging–discharging behavior of Femto-Cu relative to neat Cu foam, a comparison of the GCD curves of both at 3 mA/cm² current density is presented in Figure 4d. The Femto-Cu electrode exhibits a much longer discharge time, consistent with a C_{sp} of 215.3 mF/cm², which is more than $10\times$ that of the neat Cu foam (20.3 mF/cm²). Furthermore, the GCD cycling stability of the prepared electrode is evaluated at a current density of 10 mA/cm² over 10,000 cycles (Figure 4e), demonstrating excellent durability, retaining $\sim 87.7\%$ of its initial capacitance. The slight initial drop in C_{sp} , followed by stable charging and discharging performance even after 10,000 cycles, indicates good structural integrity of the prepared electrode. Finally, the EIS analysis is performed for both foams, and Figure 4f presents the corresponding Nyquist plots. In the high-frequency range, the Femto-Cu electrode displays a smaller semicircle than the bare Cu foam, indicating lower charge-transfer resistance. In contrast, the steeper slope in the low-frequency range suggests improved ion diffusion in the Femto-Cu sample [86–88]. This confirms that the fs laser-ablated Cu foam has enhanced electrical

conductivity and faster electrochemical kinetics due to the presence of active Cu oxide nanospheres on the Cu foam.

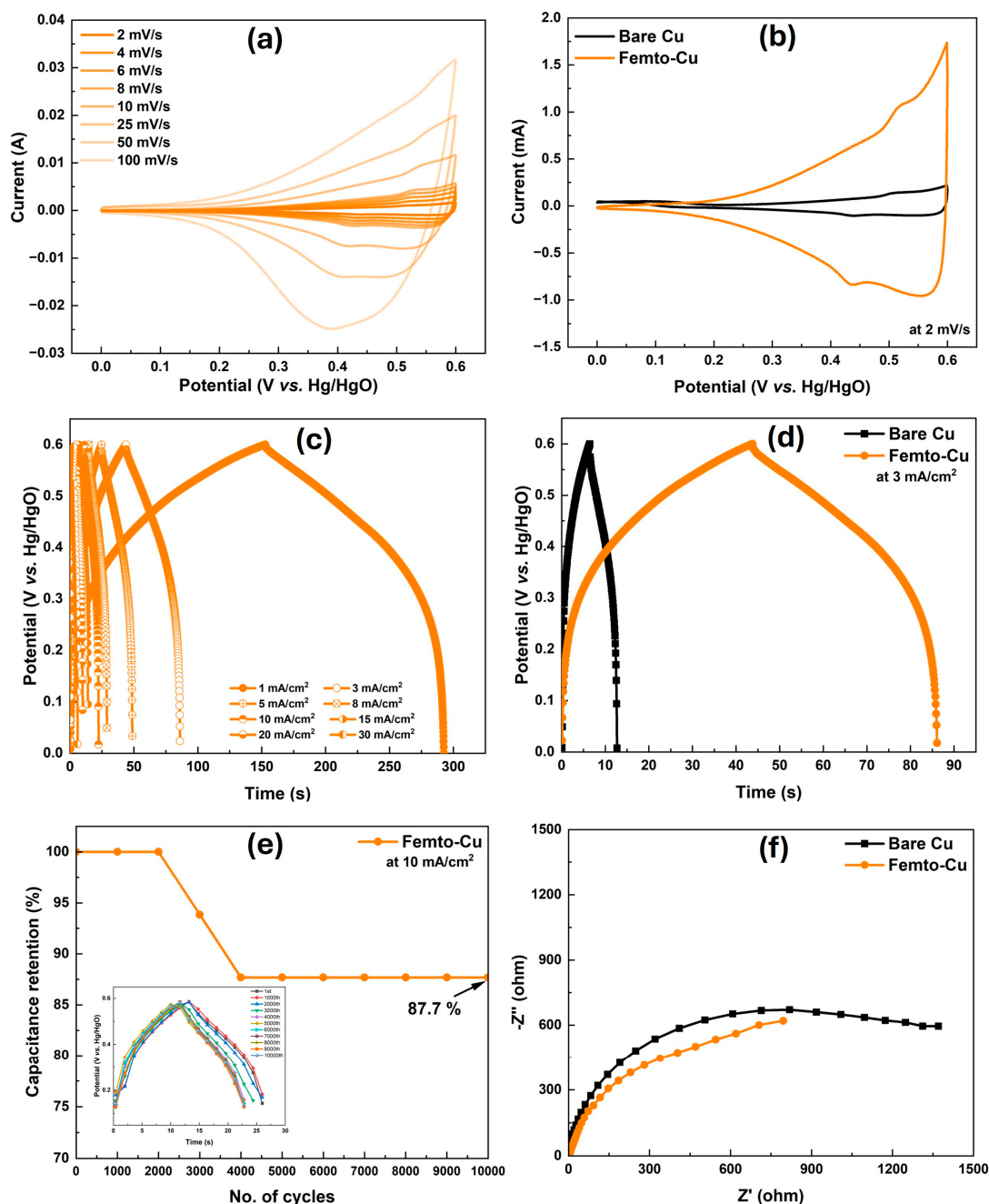


Figure 4. (a) CV recorded at various scan rates of Femto-Cu electrode. (b) CV noted at 2 mV/s scan rate of bare Cu and Femto-Cu foams. (c) GCD curves obtained at various current densities of Femto-Cu electrode. (d) GCD curves noted at 3 mA/cm² current density of neat Cu and Femto-Cu foams. (e) Capacitance retention observed up to 10,000 GCD cycles at 10 mA/cm² for Femto-Cu electrode. (f) EIS Nyquist plot of bare and Femto-Cu foams.

Power law, Trasatti's, and Dunn's methods are applied to the measured data to gain deeper insight into how the fs laser-induced Cu oxide nanospheres on the Cu surface govern the charge-storage behavior. The power law (Equation (S3)) [89] was used to obtain the b-values of the Femto-Cu foam during oxidation and reduction, and the resulting plot is shown in Figure 5a, yielding 0.82 and 0.86, respectively, indicating a mixed charge-storage mechanism. This is directly linked to the presence of Cu oxide nanospheres: their

surfaces enable rapid capacitive reactions, while their porous, interconnected structures allow electrolyte ions to diffuse into the bulk, thereby contributing to a diffusion-controlled process. The slightly higher b -values for the reduction process suggest that the reduction reactions are more surface-dominated, likely due to the easier electron transfer at the oxide–electrolyte interface during redox reactions (see Figure 3). Trasatti’s method [90] is applied to deconvolute the total stored charge using Equation (S4). The plots in Figure 5b,c were used to perform this analysis, from which the total charge was determined to be 285.71 mF/cm^2 . This total charge consists of a 113.22 mF/cm^2 capacitive contribution and a 172.49 mF/cm^2 larger diffusion-controlled component. This shows that while the bulk of the Cu oxide nanospheres participate in the redox reactions, a substantial fraction originate from the surface redox activity and double-layer charging on the nanosphere shells and LIPSS.

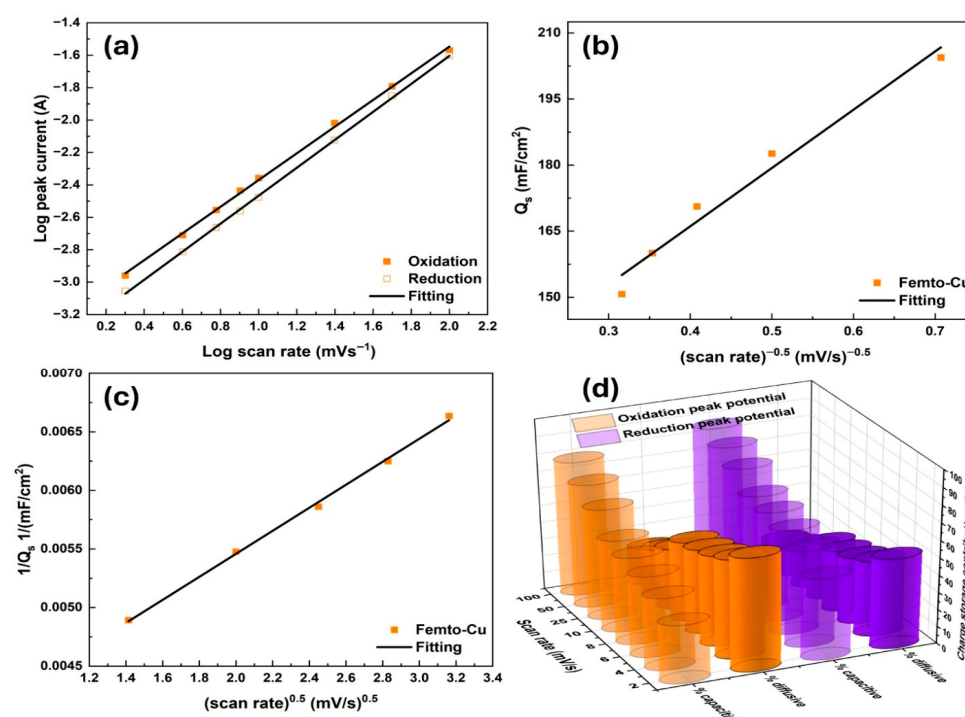


Figure 5. (a) Fitted curves for “ b ” values. (b,c) Fitted curves for total charge storage calculations. (d) Percentage charge storage contribution plot.

Dunn’s method from Equation (S5) [91] is used to plot the charge-storage contribution graphs at the peak (oxidation and reduction) potentials at different scan rates, as shown in Figure 5d, while the computed values are stated in Table 2. At low scan rates (2 mV/s), diffusion dominates (up to 65.92% for oxidation peak potential), as ions have adequate time to penetrate deeper into the Cu oxide nanospheres. But, at 100 mV/s , the capacitive contribution increases significantly—i.e., 78.52% for oxidation peak potential, which shows that the charge is mainly constrained to the outer surface of the oxide nanospheres as the ions have less time to penetrate and only stick near the surface—and well correlates with the previously observed methods and mechanisms.

Table 2. Percentage charge storage contribution at different scan rates.

Scan Rate (mV/s)	Oxidation		Reduction	
	% q capacitive	% q diffusive	% q capacitive	% q diffusive
2	34.08	65.92	47.56	52.44
4	42.23	57.77	56.19	43.81
6	47.24	52.76	61.10	38.90
8	50.83	49.17	64.46	35.54
10	53.61	46.39	66.97	33.03
25	64.63	35.37	76.23	23.77
50	72.10	27.90	81.93	18.07
100	78.52	21.48	86.51	13.49

4. Conclusions

In this work, a simple, effective, and single-step fs laser-based approach is demonstrated to fabricate the binder-free Cu oxide nanospheres directly on Cu foam, resulting in a highly active electrode for supercapacitor application. The fs laser-induced hierarchical surface of Cu foam consists of ~70 to 700 nm-sized Cu oxide nanospheres with LIPSS, which boost the electrochemical performance by offering more active sites for redox reactions. As a result, the Femto-Cu electrode delivers higher C_{sp} up to 243 mF/cm² at 1 mA/cm² than the bare Cu foam—almost 10 times more, while maintaining ~87.7% charge storing ability even after 10,000 charging–discharging cycles. Furthermore, power law, Trasatti’s, and Dunn’s methods are used to understand the kinetics and charge-storage mechanism in depth. This study revealed a mixed charge storage mechanism with a dominant diffusion-controlled contribution alongside a capacitive component, confirming the effective utilization of both surface and bulk redox processes. In conclusion, this study highlights the strong potential of fs laser processing as a single-step, binder-free strategy for designing high-performance electrode materials with enhanced stability for high-efficiency energy storage devices.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/surfaces9020043/s1>, File S1: Characterization and electrochemical equations.

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Data Availability Statement: Upon reasonable request, data can be provided by the corresponding author.

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Conflicts of Interest: There are no known conflicts of interest.

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