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# ACOUSTIC BUBBLES MICROSTREAMING-INDUCED FRAGMENTATION OF RAMIFIED IRON ELECTRODEPOSITS IN A MICROCHANNEL

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## ABSTRACT

We report on the use of a high aspect ratio microchannel (Hele-Shaw's cell) in which ramified iron deposits are electrochemically formed and next subjected to acoustic vibrations in order to fragment them into fine particles. Both the electrodes and a piezoelectric element are integrated in the microchannel. High speed visualization allows to highlight the key role of the co-produced H<sub>2</sub> bubbles (during the electrodeposition step) which generate microstreaming when subjected to vibrations. The resulting flow pattern sets in motion the ramified iron branches and bring them to bubbles surfaces where they are fragmented into submicrometric particles of iron.

**KEYWORDS:** Microstreaming, Bubbles Surface Deformations, Electrodeposition, Fragmentation

## INTRODUCTION

Microstreaming induced by oscillating bubbles subjected to acoustic waves is a well-known phenomenon which is used in microfluidic systems to realize specific operations ([1]) such as particles manipulation, micromixing, micropumping and also deformation/rupture of lipid membranes [2]. Here, we report that the mechanical stresses generated by acoustic microstreaming in a microchannel are also able to efficiently fragment metallic ramified branches made of electrodeposited iron.

## EXPERIMENTAL

The microchannel used consists of two 50 μm thick metallic plates (iron or copper electrodes) separated by 9 mm and assembled between a glass plate and a PZT disk (lead/zirconate/titanate piezoelectric, resonance frequency=4 kHz), Figure 1. A typical experimental sequence begins by applying a constant current to electrochemically form the metallic branches arranged in a columnar morphology, Figure 2a. Next, the PZT is activated and the fragmentation process is visualized by a high speed camera, Figures 2b-d. A specific image processing is applied to access the quantity of unfragmented branches as a function of time (Figure 3).

## RESULTS AND DISCUSSION

The formation of metallic branches inside the microchannel is achieved using two different iron salts as precursor for metallic ions reduction: FeCl<sub>2</sub> and CuSO<sub>4</sub>. Here, the main difference between these precursors is that copper presents a more noble character than iron and this induces differences in the reduction process. Indeed, using FeCl<sub>2</sub>, the reduction of the metallic ions is accompanied by the reduction of protons leading to the formation of a H<sub>2</sub> bubbles network between the branches (Figure 2a), whereas using CuSO<sub>4</sub> no bubbles are formed. When activating the PZT without bubbles (copper), the fluctuations of the microchannel depth (PZT vibration) induce a global fluid motion inside the microchannel not strong enough to totally fragment the metallic branches. In comparison, when bubbles are present (iron), branches disappear quickly and totally, see Figure 3. A typical fragmentation scene, as shown in Figures 2b-d, reveals that branches are transported close to a bubble surface (by a vortex flow of the microstreaming pattern [3,4]) where they are fragmented into fine particles forming a "cloud". The compressibility of the bubbles allows them to contract/dilate that induces high bubbles surface velocities [3]. Close to the bubbles surface, the resulting viscous shear stress is responsible for the fragmentation of the iron branches. However, such a significant fragmentation is only observed when the bubbles oscillate with surface deformations. We have experimentally characterized the occurrence of this non-linear regime as function of the voltage, the frequency and the bubbles size. Keeping a constant frequency, maximum surface velocities for bubbles of same size, are plotted as a function of the voltage in Figure 4; the velocity increases linearly for voltages <190 V (quasi-static displacements of the surface) and it could be increased up to 4 times, reaching 4 m/s when surface deformations occur.

This significant velocity increase allows to reach required viscous shear stresses to fragment the branches. The produced-particles size distribution (measured with a morphological optical analyzer Morphologi G3, Malvern) shows a significant proportion of submicrometric particles (not shown).

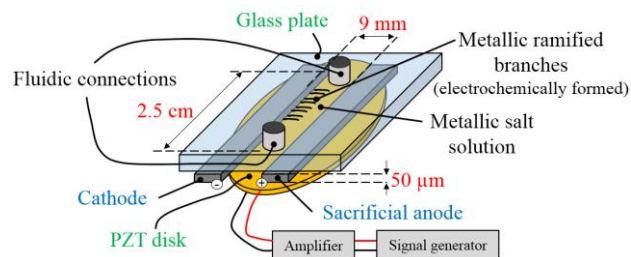


Figure 1: sketch of the experimental set-up

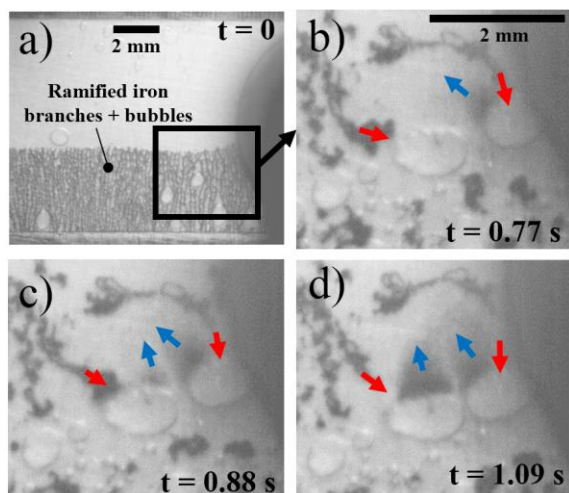


Figure 2: a) visualization of the ramified iron branches after the growth step and just before the start of the fragmentation ( $[FeCl_2]=0.1 M$ ,  $I=1 mA$ ), b-d) magnified views of a fragmentation scene by oscillating bubbles; red arrows: motion of the branches towards bubbles surface; blue arrows: expulsion of a cloud of particles ( $V_{pp}=122 V$ ,  $f=4 kHz$ , square wave, 1500 FPS)

## CONCLUSION

Electrochemically formed iron branches could be fragmented into submicrometric particles in a vibrating and a high aspect ratio microchannel if bubbles are present and if the applied voltage ( $V_{pp}$ ) is sufficiently high to induce bubbles surface deformations. The fragmentation of the metallic branches are due to the fast deformations of the instable bubbles surface.

## ACKNOWLEDGEMENTS

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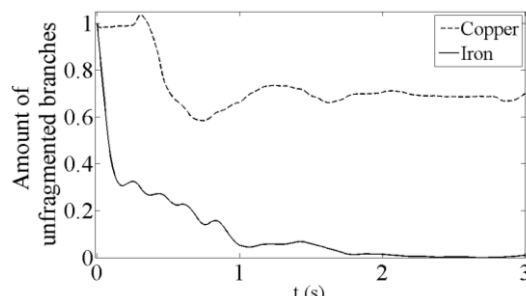


Figure 3: amount of unfragmented branches as a function of time for copper and iron branches ( $[FeCl_2]=[CuSO_4]=0.1 M$ ,  $I=1 mA$ ,  $V_{pp}=254 V$ ,  $f=4 kHz$ , square wave, 1500 FPS)

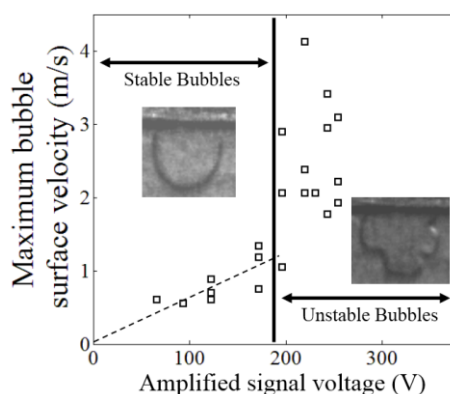


Figure 4: maximum bubble surface velocity measured for bubbles of same size ( $600 \mu m$ ) as a function of the voltage supplied to the PZT ( $f=4 kHz$ ); velocities are obtained measuring surface displacements on magnified visualizations of bubbles such as those shown as insets