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Evolution of model roughness on quasi-pure aluminum during plasma electrolytic polishing

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ABSTRACT

The present paper studies the material removal evolution, during Plasma Electrolytic Polishing (PeP), on model roughnesses prepared on 1050 aluminum alloy (AA), i.e. a quasi-pure aluminum, used as a model substrate.

The results obtained in this study confirm the significance of the applied voltage on aluminum removal. In particular, the minimal voltage to be applied to generate the plasma is seen to be above 230 V. Meanwhile, at a higher voltage of 300 V, high quality polishing is obtained. Using this technique, rolling streaks are eliminated rapidly without the workpiece's geometry being adversely affected.

Finally, roughness evolution was accurately studied on a series of samples with various well-defined levels of model roughness generated by microindentation. The study shows that only a few minutes of processing are needed to obtain a significant reduction in roughness, even representing up to 70%. Results show also that roughness valleys do not undergo material removal at all as long as peak to valley height remains high enough. As the process goes on, roughness (i.e. mainly peaks) is reduced and valleys become submitted to material removal as well.

1. Introduction

Various polishing processes can be applied for metal parts, with chemical, electrochemical and mechanical polishing now being the most commonly used, especially for parts with large surfaces. Sandblasting provides a rapid and easy solution but is not effective in polishing parts with complex shapes. Meanwhile, such more or less complex shapes can be treated by liquid phase polishing (i.e. electropolishing and chemical polishing). However, these two processes have two main drawbacks. Firstly, this method is time consuming, sometimes lasting several hours. Secondly, the electrolytes used are dangerous for operators and represent an environmental hazard.

Polishing techniques can also be combined to treat the same workpiece. However, the present paper focuses on an innovative surface treatment known as Plasma Electrolytic Polishing (PeP). PeP is based on the formation of a thin vapor skin resulting from the vaporization of the aqueous electrolyte at the metal surface. Applied voltages are sufficiently high to induce the ionization of the elements present in this vapor skin and thus a plasma is formed, inducing material removal and thus polishing of the part.

PeP can be applied to produce very smooth and shiny surfaces within

a short treatment time (a few seconds to 10 min). In addition, the electrolytes used for PeP treatment generally comprise at least 80% by mass of water, making it an advantageous alternative to the use of conventional electropolishing electrolytes. Finally, the technique appears not to encounter any technical problems in uniformly treating parts with complex shapes.

The technology was already studied on various metals and alloys, e.g. stainless steels [1–18], titanium alloys [19–22], CoCr alloys [22,23], copper [24], brass [23], magnesium [25] and WC-Co [26]. Some papers have investigated this process with respect to aluminum substrates. Previous studies thus used unspecified aluminum substrates for Duradji et al. [24,27], AA 2017 for Kaputkin et al. [16], and other alloys: AMG (i.e. AA 5XXX), D-1 (i.e. AA 2098), D-16 (i.e. AA 2024) and D16P (i.e. AA 2094), AMTs (i.e. AA 3XXX), AD33 (i.e. AA 6061), AD35 (i.e. AA 6082) [28]. Therefore, previous studies about Aluminum PeP, were performed on aluminum alloys, i.e. multiphased materials showing different initial roughness.

Moreover, according to Duradji [24,27], the applied voltage is the most significant parameter since it controls PeP initiation and determines the final quality of polishing. Their results show in particular that there is an optimum voltage range (300–350 V for unspecified

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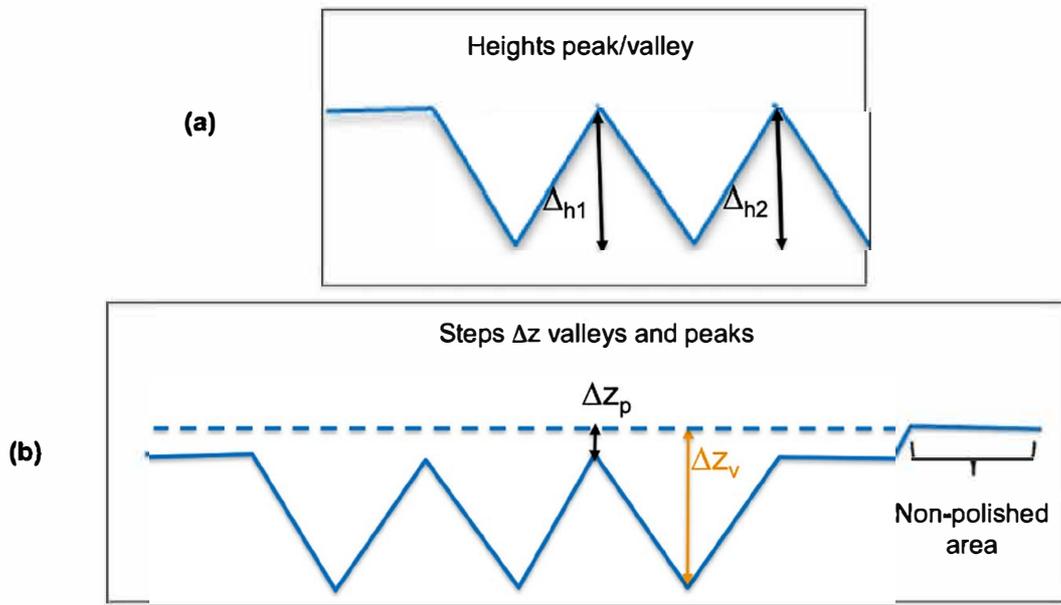


Fig. 1. Diagrams of the different measurement parameters made on the indentations: (a) peak to valley heights; (b) step heights between an unpolished area and the top of the peaks or the bottom of the valleys.

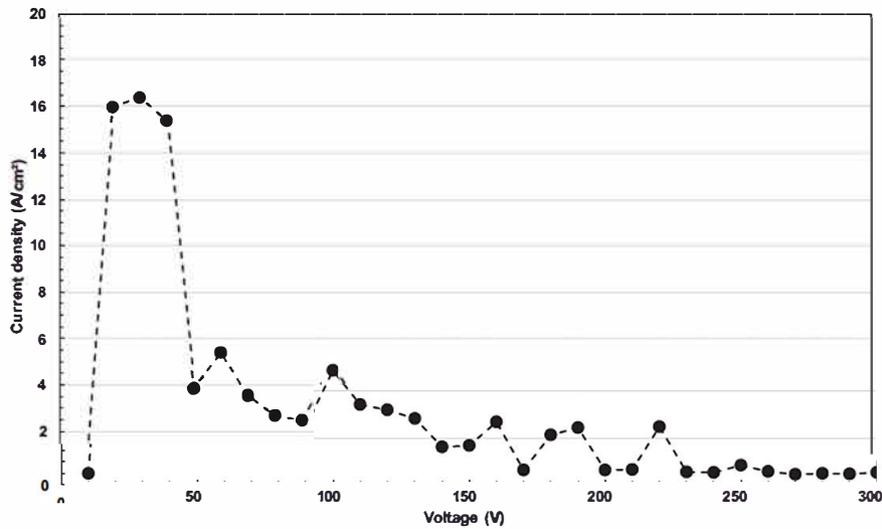


Fig. 2. Evolution of the current density (A/cm^2) as a function of the applied voltage (V) on an anode made of AA 1050 wire (0.20 cm^2) immersed in the electrolyte at $60\text{ }^\circ\text{C}$.

aluminum) below which the plasma is difficult to initiate and the vapor skin is unstable around the electrode. Above this voltage range ($> 400\text{ V}$), polishing quality tends to become degraded, with an increase rather than a reduction in roughness.

The present study's intention is to study evolution of model roughness profiles prepared on a quasi-pure aluminum substrate (i.e. AA 1050, with 99.5 w% Al minimum), in order to avoid the influence of composition inhomogeneity and multiphases microstructures and to finally study precisely aluminum removal during the PeP process.

First, we study, in our experimental conditions, the influence of the applied voltage on both plasma stability (via current response) and

polishing quality (i.e. surface state and geometric integrity). Secondly, we propose an original method in order to create a model roughness on quasi-pure aluminum substrate. To do so, a row of five consecutive pyramidal indents of controlled depth and geometry are created by Vickers indentation, effectively leading to a series of "valleys" and "peaks" with precisely controlled topography. Using 3D interferometry, this model roughness allows to follow precisely where and when material is removed during PeP process, depending purely on the initial surface topography.

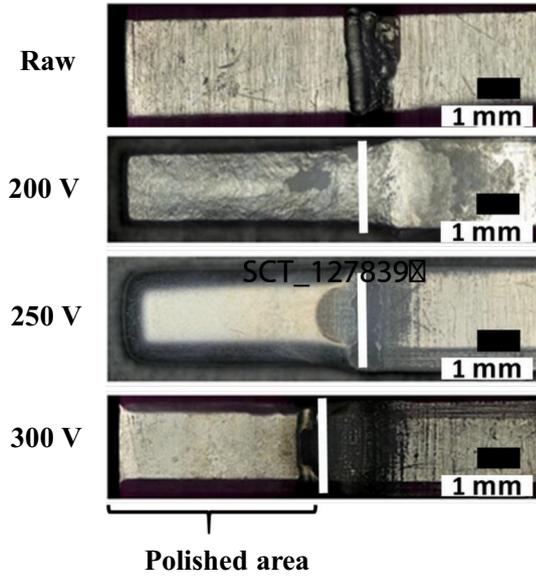


Fig. 3. Optical microscopy images of samples after 3 min of polishing at three different voltages.

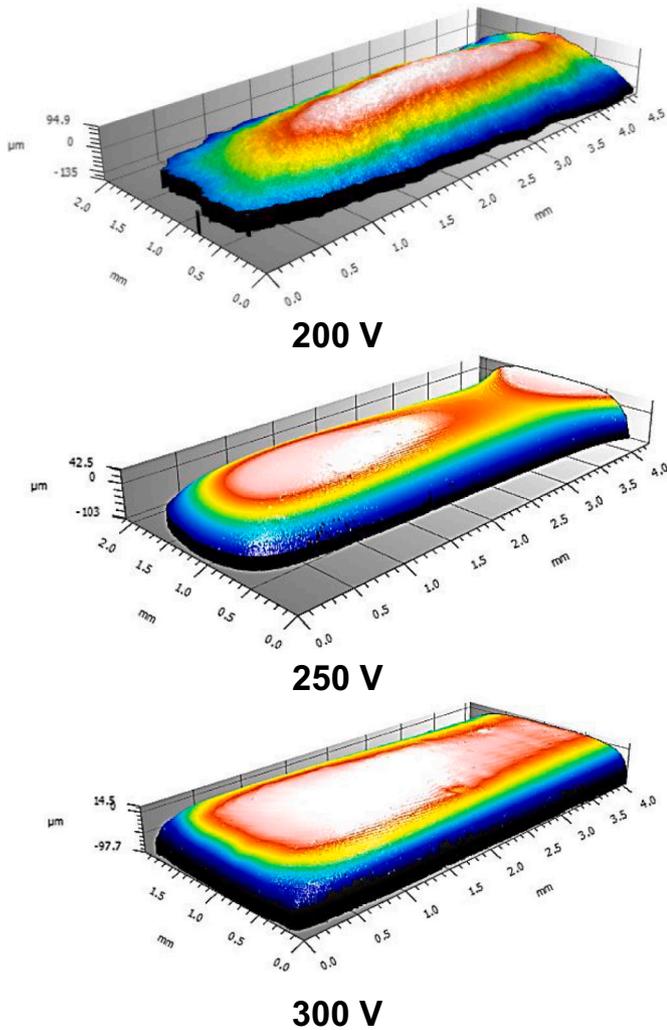


Fig. 4. 3D profiles obtained by optical interferometry of samples (0.25 cm²) after 3 min of polishing at three different voltages.

2. Experimental

2.1. Material and process

The substrate material was 1050 aluminum alloy (AA). Its chemical composition in weight percent is <0.40%Fe, < 0.25%Si, < 0.07%Zn, < 0.05%Cu, < 0.05%Mg, < 0.05%Ti, < 0.05%Mn while Al accounts for the remainder. Three types of substrate were used: wires (500 µm diameter; 0.20 to 0.25 cm² of immersed surface), thin strips (3 mm width, 1 mm thick, 4 mm of immersed depth for a treated surface of 0.25 cm²) and large strips (1 cm width, 1 mm thick, 2.5 cm of immersed depth for a treated surface of 5 cm²).

The experimental setup for the PeP treatment was based on those described in the bibliography [3,23] for the treatment of other metals. This consisted of a five-liter beaker (a single-wall glass beaker) into which three to four liters of electrolyte (oxalic acid 3w%, ammonium chloride 10w%, potassium chloride 4w% [27]) introduced and heated initially to 60 °C. The workpiece (i.e. the anode) was surrounded by two counter-electrodes, made of platinized titanium grids (160*120 mm, i.e. about 115 cm²), located at 4 cm on both sides. Finally, the generator used was a TDK lambda 1500 model, which can deliver a maximum of 5 A and 300 V for a power of 1500 W. Experiments were conducted in the 20–300 V voltage range, while processing times ranged from 15 s to 10 min.

2.2. Characterizations

Surface views were taken using either optical microscopy (Keyence 3D VHX-1000) or scanning electron microscopy (JEOL JSM-6510LV).

Surface roughness was studied by means of an interferometric 3D microscope in Vertical Scanning Interferometry (VSI) mode, allowing surfaces with significant height discontinuities to be measured. The equipment used was an S-Neox microscope from Sensofar with a ×10 lens (scanned surface 1754 × 1320 µm) or ×50 (scanned surface 351 × 264 µm) of the Mirau type. The norm ISO25178 determined how measurements of roughness parameters were obtained. The interferometric microscope produced digital data maps of the surface topography that usually required subsequent processing. The roughness value, Sa, was thus measured by applying two filters:

- A “Restore” filter was used to interpolate points that could not be measured. The missing “z” altitude of these points was calculated by averaging the “z” altitude of the 8 points around the vacant point. It is important to note that less than 10% of the data were thus corrected. This was the minimum requirement for the measurements to be validated.
- A “Form Removal” filter allowed the tilt of the microscope stage or the curvature of the test sample to be corrected.

Having applied these two filters, the Sa roughness parameter was extracted. The Sa roughness parameter is the arithmetic mean of the absolute value for the height in a sampling area:

$$S_a = \frac{1}{A} \int_A |z(x, y)| dx dy \quad (1)$$

The roughness parameters were always measured on three areas spaced by 5 mm at the center of one face of each sample.

Model roughness defects were artificially created, thus offering reproducible shapes to be studied accurately. To do so, series of peaks and valleys of different sizes were generated thanks to a Vickers microindentation machine. To create defects of different sizes, different masses (100, 400, 500 and 600 g) were applied to the Vickers type indent. The untreated part of the specimen was masked by an electrically insulating and chemically resistant polymer protection (SOCO-MASK PRT 2130).

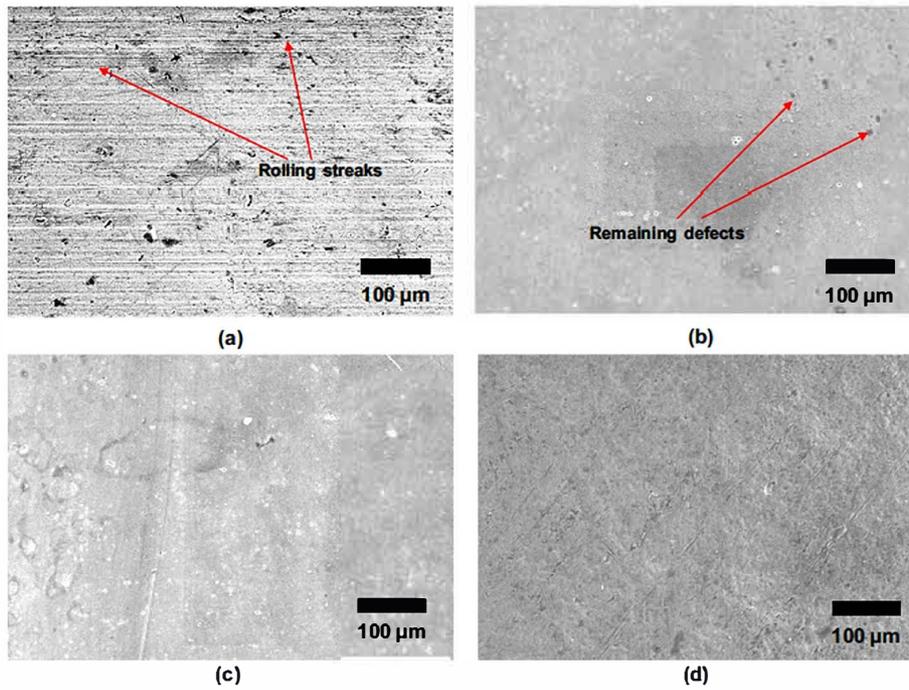


Fig. 5. SEM surface views of samples (0.25 cm^2) (a) raw, and after (b) 30 s, (c) 60 s and (d) 150 s of polishing at 300 V.

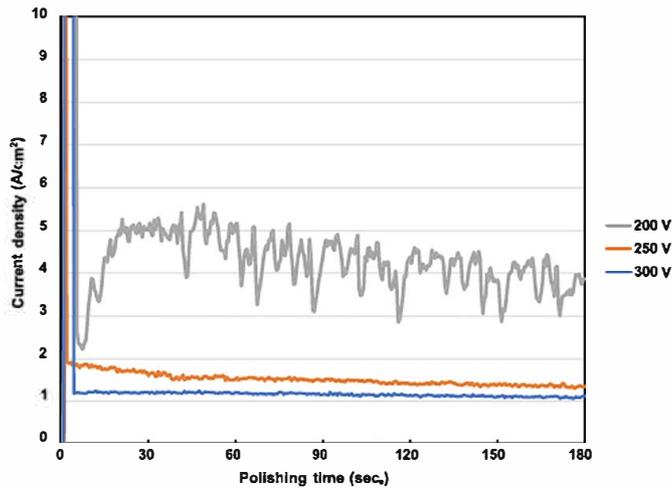


Fig. 6. Evolution of the current density for different applied voltages.

After PeP treatment, the peaks and valleys (Δz_p , Δz_v) were measured according to the following procedure (Fig. 1 (a)): the protective mask was first removed so as to be able to measure steps between the unpolished area and the various indentations. For each series of indentations, a first step was measured with the top of each peak Δz_p and another step was measured with the bottom of each valley Δz_v (Fig. 1(b)).

3. Results and discussion

3.1. Specific PeP treatment on AA 1050

A first study on AA 1050 wire was conducted to determine the

voltage range within which the plasma (and therefore the PeP treatment) is triggered under these operating conditions. A curve indicating the change in current density as a function of the applied voltage is shown in Fig. 2. The behavior is similar to that obtained by Kellogg [29] on platinum wire and is divided into three specific zones.

In the first zone (A to C), i.e. for low applied voltages from 10 to 50 V, there is a peak of current that causes significant electrodisolution of the anode. Then, in the second zone (C to D) for applied voltages between 50 and 220 V, there are current fluctuations, associated with a partial formation of the vapor skin, with the anode showing zones still in contact with the electrolyte. In zone (D to E), between 230 and 300 V, the plasma extends along the length of the wire and the current, now stable, is about 0.1 A. The voltage suited to instigate PeP thus seems to be above 230 V. In addition, the vapor skin thickness is estimated to be about $250 \pm 22 \mu\text{m}$ looking at a number of images taken during the treatment.

The influence of the applied voltage was then studied on rolled sheet, with the treated surface being 0.25 cm^2 . The first optical observations (Fig. 3) show that polishing is of poor quality at an applied voltage of 200 V, with the geometry and surface condition of the sample being totally degraded. At 250 V, the rolling streaks are eliminated and enhanced brightness is found. However, the geometry is significantly impacted, with considerable loss of material and a rounding of the angles. As for 300 V, the surface condition was improved with the disappearance of rolling streaks and few modifications to angular geometry.

The 3D profiles for these same surfaces (Fig. 4) also show modification of angles at 200 and 250 V. Moreover, these profiles clearly confirm the absence of rolling streaks after 3 min of polishing at 250 V and 300 V. The SEM images of the surface before and after polishing at 300 V are presented in Fig. 5. These pictures also confirm the disappearance of rolling streaks as from 30 s of polishing (Fig. 5a and b). Some defects (i.e. micrometric holes) remain after 30 and 60 s (Fig. 5b and c), but an extended polishing time (150 s) allows a smooth surface with few defects to be obtained (Fig. 5d).

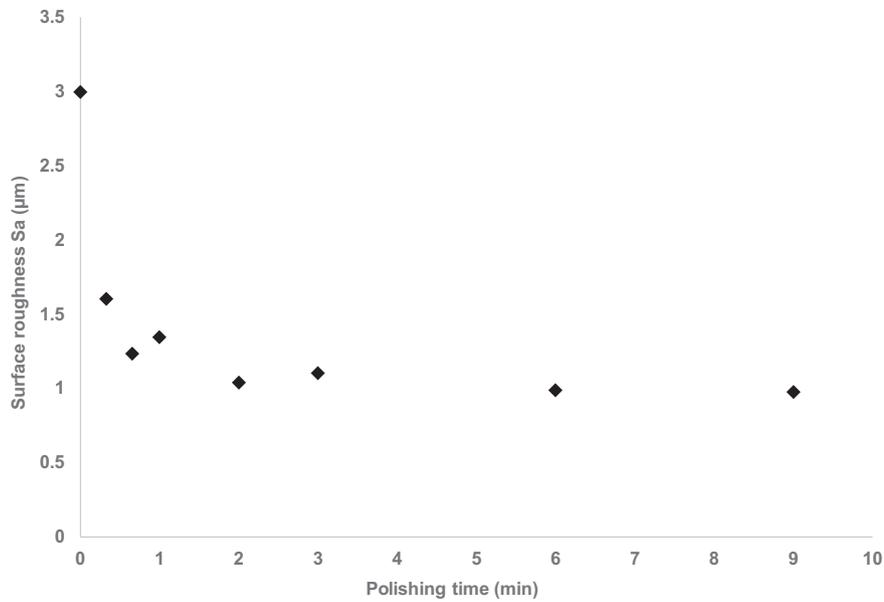


Fig. 7. Evolution of surface roughness parameter S_a with polishing time at 300 V on samples (5 cm^2) with an initial roughness increased to $3 \mu\text{m}$.

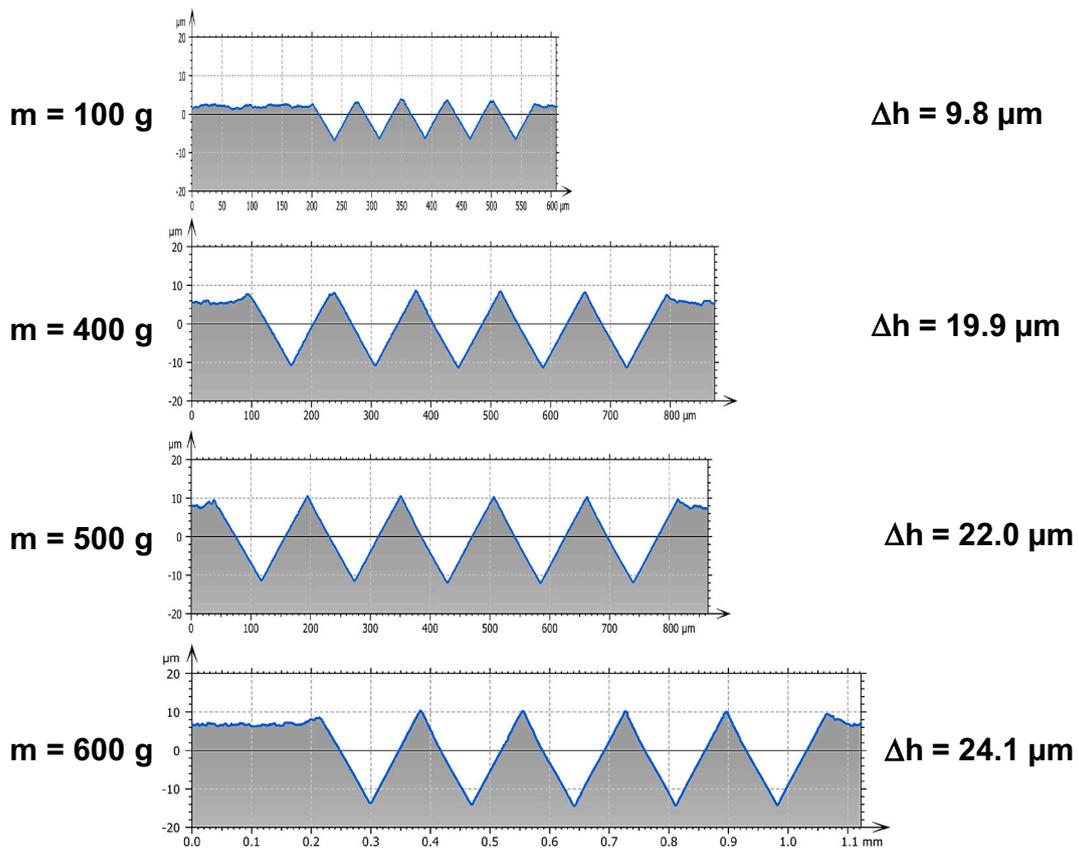


Fig. 8. Profiles obtained by optical interferometry of four sets of indentations before polishing.

Fig. 6 shows the changes in the current density for different imposed voltages (200 V, 250 V and 300 V). The behavior observed appears similar to that observed in Wang's study [30]. In general, the intensity

decreases as the applied voltage increases. At 200 V, the current remains unstable throughout the duration of treatment. This is due to the fact that the vapor skin is unstable and the electrolyte periodically comes

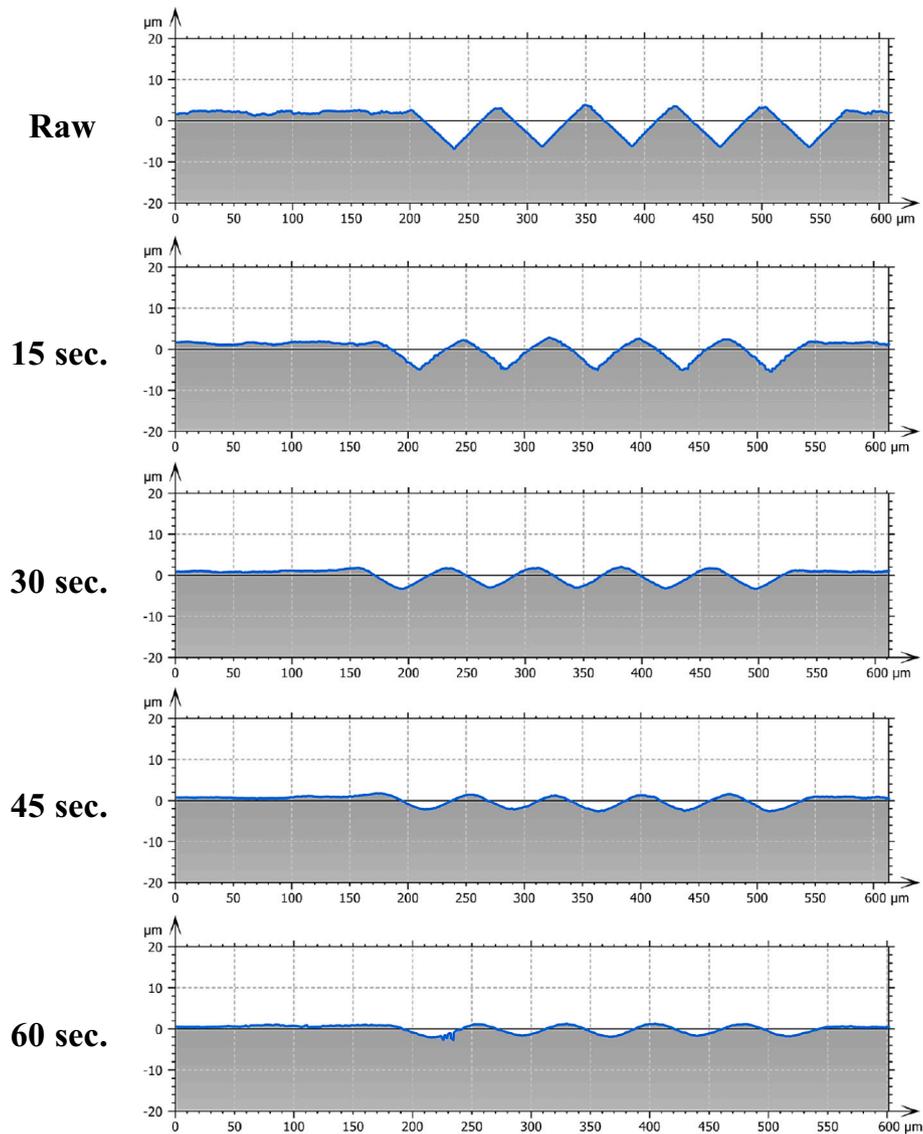


Fig. 9. Evolution of indentation shape with polishing duration at 300 V for Vickers indentations obtained with $m = 100$ g.

into contact with the anode (see the C–D portion of the curve (Fig. 2)). At 250 and 300 V however, the current remains stable. It seems that for these two voltages, the vapor skin, and thus the plasma, is stable and covers the entire anode surface.

The study then went on to consider the influence of the PeP processing time at 300 V. For an initial roughness of $S_a = 2.61 \pm 0.25 \mu\text{m}$ (obtained by prior abrasion with 180 SiC paper), the roughness rapidly decreases monotonously with the processing time (Fig. 7), to reach a final value of about $0.98 \pm 0.19 \mu\text{m}$, corresponding to a roughness reduction of 62%.

To summarize, experiments on AA 1050 wire and rolled sheet demonstrated the relevance of the applied voltage with respect to PeP quality, and a minimum plasma initiation voltage above 230 V was demonstrated. Below this voltage, no effective polishing was observed on the aluminum alloy, while with applied voltages of 250 and 300 V, the PeP process was successful.

3.2. Evolution of model roughness

The indentation profiles before PeP obtained by optical interferometry are shown in Fig. 8, with the x-axis and y-axis scales being identical for all these profiles so that these different defects can be readily compared. For an applied mass of 100 g (the lowest), the peak/valley mean height (Δh) is $9.8 \mu\text{m}$, while for the highest applied mass (600 g) Δh is $24.1 \mu\text{m}$.

For PeP performed at 300 V, the change over time (15, 30, 45 and 60 s) of the indentations profile for an applied mass of 100 g is shown in Fig. 9. Profiles corresponding to the other masses can be found in Figs. 10, 11, 12). On all these profiles, it emerges that the tops of the peaks become rounded after 30 s of PeP. Furthermore, after 60 s of treatment, defects of up to $10 \mu\text{m}$ in height are almost entirely removed by the PeP under such conditions.

Fig. 13 summarizes the peak/valley height (Δh) reduction for all sizes of defects in relation to polishing time. This peak/valley height

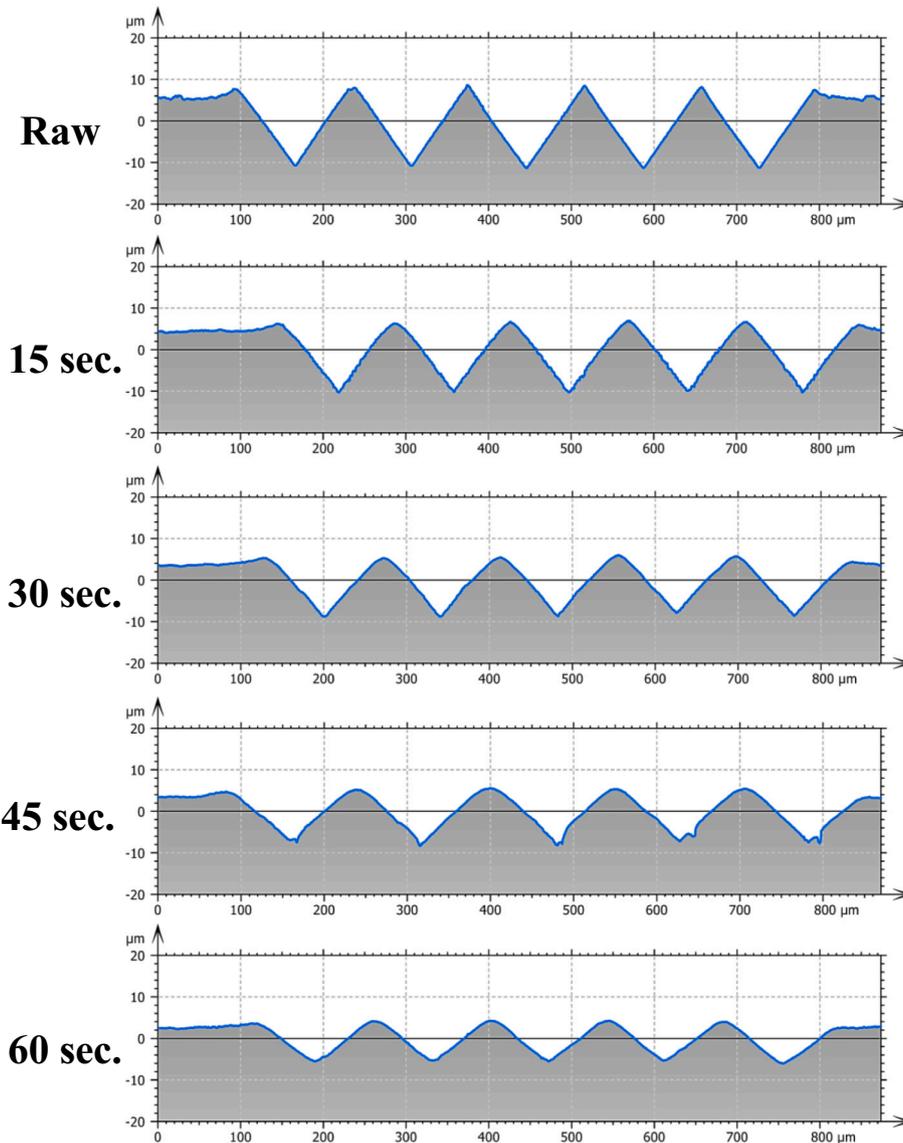


Fig. 10. Evolution of indentation shape with polishing duration at 300 V, for Vickers indentations with $m = 400$ g.

diminishes monotonously over time for all defects. For the largest defects ($m_{\text{applied}} = 400$ to 600 g), the decrease is 53, 50 and 44% after 60 s of PeP. For the same treatment time, the reduction reaches up to 70% of the height of small defects ($m_{\text{applied}} = 100$ g). PeP thus allows “small” defects to be polished faster than “large” defects.

In addition, measurements of steps between peaks and valleys (Fig. 14) show that the peaks of the various defects undergo a monotonous material removal over time. Removal of material from the peaks (Fig. 14) is comparable for the four defect sizes, since after 60 s of PeP, step values Δz_{peak} were very close. However, material removal behavior is not the same for the valleys (Fig. 14). For large defects ($m_{\text{applied}} = 400$ to 600 g), the valley steps change little over time during PeP. However, for smaller defects ($m_{\text{applied}} = 100$ g), the step Δz_{valley} increases significantly with the PeP duration. It is readily observable on the profiles for $m_{\text{applied}} = 600$ g (Fig. 12) that the geometry of the peaks is more greatly impacted over time, as compared with the geometry of the valleys.

If it is clear that material removal is always faster on peaks than on valleys, inducing indeed a polishing effect over time, those results show that valleys remain entirely unaffected only beyond a certain depth, at least on those model pyramidal shaped defects and on quasi-pure aluminum alloy. This conclusion may arguably not be extended to multiphases alloys such as AA2024 where composition inhomogeneity can have an important impact on material removal [5,7,8,16]. Furthermore, the pyramidal shape of those defects, even if providing interesting information on material removal during PeP, are quite different from the roughness shape observed on materials being obtained by either conventional or additive manufacturing. Nevertheless, those results show that, depending on experimental conditions, PeP can induce, to a lower extent, material removal in the valleys of roughness, even if the plasma discharge is triggered on peaks where the electric field is higher.

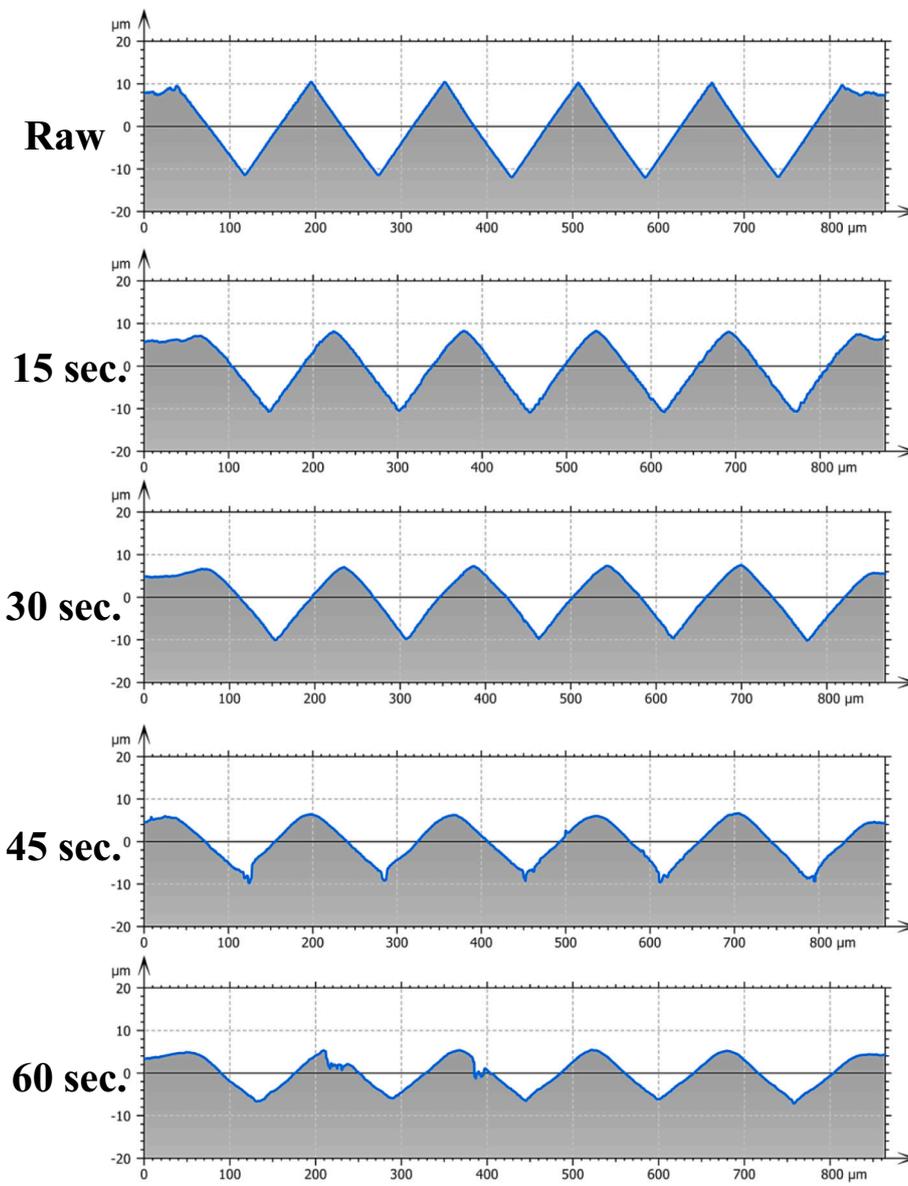


Fig. 11. Evolution of indentation shape with polishing duration at 300 V, for Vickers indentations with $m = 500$ g.

4. Conclusion

PeP experiments were performed on a quasi-pure aluminum (i.e. AA 1050), confirming the significance of the treatment time and of the applied voltage. Under these experimental conditions, the voltage to be applied to generate a plasma was seen to be above 230 V.

At a polishing voltage of 300 V, the polishing is of good quality, meaning that rolling streaks generated during rolling are rapidly eliminated without any modification to the workpiece's geometry. Following this, material removal was studied on model roughness of different sizes generated by microindentation. In particular, the results show that roughness peaks undergo material removal while the deepest valleys remain unaffected by the process. As the process goes on though, peak to valley height decreases and valleys start to undergo material removal as well. A useful follow-up to the present study will be to investigate

polishing of parts from additive manufacturing or those with more complex shapes.

CRediT authorship contribution statement

A. Spica: Conceptualization, Methodology, Investigation, Writing – original draft, Visualization. **J. Roche:** Conceptualization, Methodology, Validation, Investigation, Writing – original draft, Writing – review & editing, Supervision, Visualization. **L. Arurault:** Conceptualization, Methodology, Validation, Writing – original draft, Writing – review & editing, Supervision, Visualization, Project administration, Funding acquisition. **M. Horville:** Investigation, Visualization. **J. Rolet:** Conceptualization, Project administration, Funding acquisition.

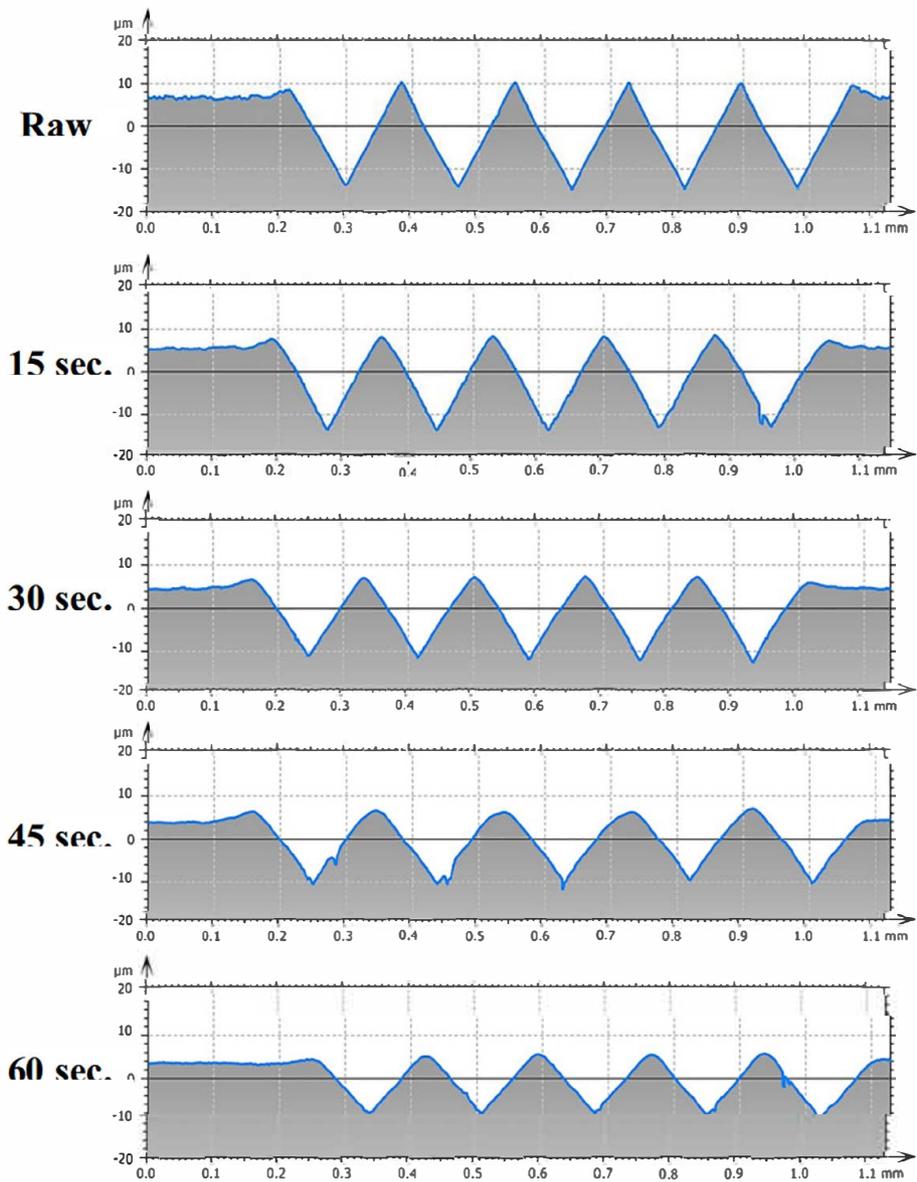


Fig. 12. Evolution of indentation shape with polishing duration at 300 V, for Vickers indentations with $m = 600$ g.

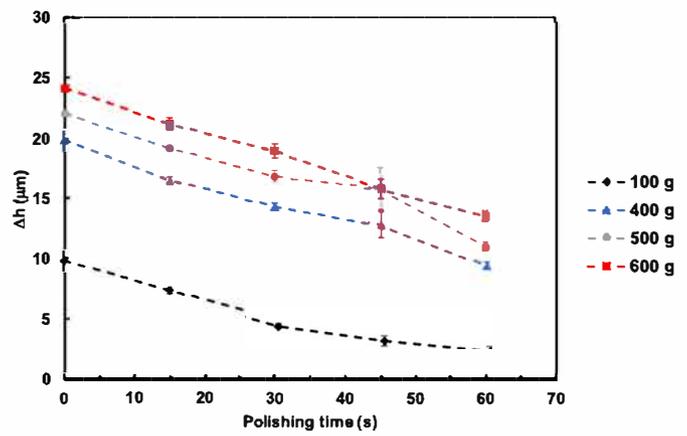


Fig. 13. Evolution of peak to valley height (Δh) for different indentation sizes as a function of the polishing duration.

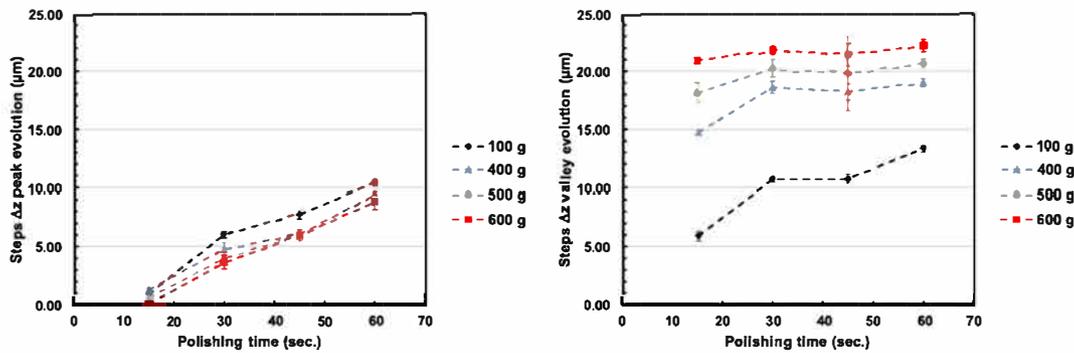


Fig. 14. Evolution as a function of the polishing duration of the steps between an unpolished zone and (a) the top of the peak Δz_{peak} or (b) the bottom of the valley Δz_{valley} for different indentation sizes.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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