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A NEW, ORIGINAL AND ANALYTICAL PROPOSAL FOR THE CAUSES OF THE RELATIONSHIP BETWEEN CURRENT AND LAYER BEHAVIOR AND ITS NANOSTRUCTURES ORIGINATING ON A METAL BASE

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Abstract: The relationship between the development of nanostructures and the current of charge carriers, both generated in a layer built on a metal base, titanium, for example, by anodizing engineering, or some equivalent, is still an open problem, that is, not completely solved and closed in the area of new materials (biomaterials, etc.). In this area, the standard solution used for this problem is incomplete and is based on classical theory, which sometimes reaches the semi-classical frontier. Then starting from a critique, throughout this article, a new, original, analytical and broader and improved proposal for the causes of the relationship between nanostructure development and charge carrier current is given, but grounded in the quantum electrodynamics of particle motion in the presence of an external potential (barrier potential). Thus, the treatment of experimental results in this area becomes more widely understood for applications in the manufacture of biomedical implants or other practical uses (photocatalysis, etc.).

Keywords: Current, Nanostructure, Electrodynamics, Potential Barrier, Anodization, Biomaterial

HIGHLIGHTS

• Interactions between particles determine the shapes in nanostructures and current

• The barrier potential drives the current behavior through a layer

• A layer and its nanostructures can be associated with a separate barrier potential

• Nanostructures on a layer do not always lead to oscillations in the current

INTRODUCTION

(Importance of the problem under study and recent articles)

Micro and nanostructures and charge carrier currents originating from films being built on a metal (Ti, for example) base[1] have been widely studied because they contribute to the development of new materials that are used as biomedical implants [1], 2020, [2], 2021, or new materials that are used for other purposes, photocatalysis [3], 2020, for example.

In this context, Guo *et al.* [4], 2022, performed an experimental study of the production of nanostructures and current originating from films under construction on a titanium base (Ti). In this work, they used either *single*-step or *two*-step Anodization Engineering to build the films and their nanostructures and current, but assumed to obtain interesting effects produced when considering anodizations with *fresh* electrolyte or *aged* electrolyte and the anodization proceeded in a *short time[2]*. The first step, in the *two*-step anodizing, was processed for the purpose of aging the electrolyte. Here, the anodizations occurred without the titanium substrate (Ti) and anodizations of 15 h each and 30 h each were performed by repeating the cycle under 80 V voltage. In this study, after analyzing the experimental results, they obtained: **a)** the formation of TiO_2 pore-like (nanopores:TNPs) and tube-like (nanotubes) nanostructures, **b)** that, using *fresh* electrolyte, the alignment of the nanopores occurred early, at about 120 s, compared to *aged* electrolyte, which led to the appearance of alignment just before 600 s, **c)** that, with *fresh* electrolyte, the nanopores showed more favorable wetting capacity, protein adhesion, and mechanical properties compared to the nanopores obtained using *aged* electrolyte, and **d)** measurements of current density versus anodization time (in seconds), where it was evident that a longer anodization time is required to reach dynamic equilibrium when the electrolyte is *aged*; in this case, also, the

1 Base means the surface of the metal under anodizing

2 *Short time* means of the order of 300 seconds (5 min) at most

graphs showed three distinct phases when the anodizing time is approximately 300 s, as expected; however, in the case of *fresh* electrolyte, this behavior does not occur. Thus, through the different phases of the current, they were able to suggest in which temporal phase of anodization the nanostructures manifest their developments, as stated in the review by Regonini *et al*. [5], 2013.

Mohan and collaborators [6], 2020, conducted an experimental study of the influence of different temperatures ((5, 10, 25, 30, 50 and 70) °C) and time on TiO_2 nanotube formation and current in films under construction on a titanium alloy base, Ti-6Al-7Nb, immersed in electrolyte solution from an anodizing circuit under 30 V. Thus, important results regarding the diameter, thickness and shape of TiO_2 nanotubes were achieved in the case of anodization at 25 °C. In this case, the nanotubes showed 125 nm average diameter, approximately, 250 nm length, 30 nm and 35 nm wall and inter-tube thickness, respectively. These characteristics are potentially better for ensuring a more stable geometry of nanotubes, which contribute to the fabrication of biomimetic implants [1], 2020. Also, it was evidenced that this contribution can be obtained by controlling the variation of the anodizing time. Additionally, they measured the current of charge carriers during the anodization and elaborated graphs of current versus time. Again, in order to relate the development of nanostructures, in the study case: nanopores and nanotubes, with the three phases of the graphical form of the current, as described by Regonini *et al*. [5], 2013, and others [7], 2005, [8], 2023.

Yang *et al* [9], 2019, in each anodization procedure of titanium (Ti), immersed in an electrolyte under 50 V and 30 °C, and forming a closed circuit, they introduced a different amount of NH_4F (0.2, 0.3, 0.4 and 0.5) % wt of NH_4F with the aim of studying the effect of varying this mixture[3] on the development of TiO_2 nanotubes. The result obtained was that the growth rate of the anodized nanotubes increases slowly with increasing NH_4F concentration. However, varying the voltage of (90, 80, 70, and 60) V, respectively, at the concentrations of (0.2, 0.3, 0.4, and 0.5) % wt of NH_4F, obtained the growth rate of nanotubes of (2.191, 0.863, 0.538, and 0.497) $\mu m.min^{-1}$. That is, the growth rate of the nanotubes decreases with increasing concentration and order of variation of these voltages. Thus, notice that for high voltage, 90 V, the growth rate was 2.191 $\mu m.min^{-1}$, however, for threshold voltage of 50 V, the growth rate was 0.140 $\mu m.min^{-1}$. The comparison of these last two results indicates a rapid growth of nanotubes at low NH_4F concentration, which cannot be explained the exact cause of this growth by the proposed fluoride ion dissolution reaction. This classical theory, as in the previous studies, was used to give an answer to the relationship between nanotube development and the three phases of charge carrier current behavior versus anodization time, however, it is incomplete.

For more details of the experimental procedures and results described before, it is recommended to read the referenced articles and others that may broaden and clarify the thoughts of those interested, such as Mendes *et al*. [10], 2020, Estanislau *et al*. [11], 2023, etc.

RELATIONSHIP BETWEEN NANOSTRUCTURE AND CURRENT

(Presentation and objective of the problem under study)

In Introduction, in all the experimental studies, regarding nanostructure and its development related to charge carrier current, a classical theory (dissolution reaction)

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3 This mixing means the effect of varying the concentration of the electrolyte medium

was used to explain the nanostructure development associated with the three phases of current versus anodization time behavior [4], 2022, [5], 2013, [6], 2020, [7], 2005, [8], 2023, [9]. 2019, [12], 2020, etc. But, as studied by Yang *et al.* [9], 2019, the classical dissolution reaction theory cannot give the cause for the rapid and large growth rate of TiO_2 nanotubes developed during titanium anodization under voltage above 50 V.

This problem of the relationship between nanostructure development and current behavior versus anodization time is more complex and deeper to have as a cause the proposal contained in these references, where it is in a restricted view of the problem under study. The slopes and rises of the behavior of the current of charge carriers versus anodization time, for example, as shown in part (2) of Fig. 01, necessarily do not have as a cause only the arising and development of nanostructures (nanopores, nanotubes, etc.).

Then, in this paper, the author aims to give a new and original causal proposal, more extensive and analytical[4], that improves the understanding and accuracy of the relationship between nanostructure development and current behavior of charge carriers obtained on the modified surface during anodization engineering on a metal base or one similar to it. This proposal is described in the next section and Fig. 01 represents the experimental abstraction of it.

PROPOSITIONAL RELATIONSHIP BETWEEN CURRENT AND NANOSTRUCTURE

(Discussion and arguments, a new causal and analytical proposal for this relationship)

Here, the behaviors and nature of the current density of charge carriers through a layer with its nanostructures in construction on a metal base (Ti, for example) immersed
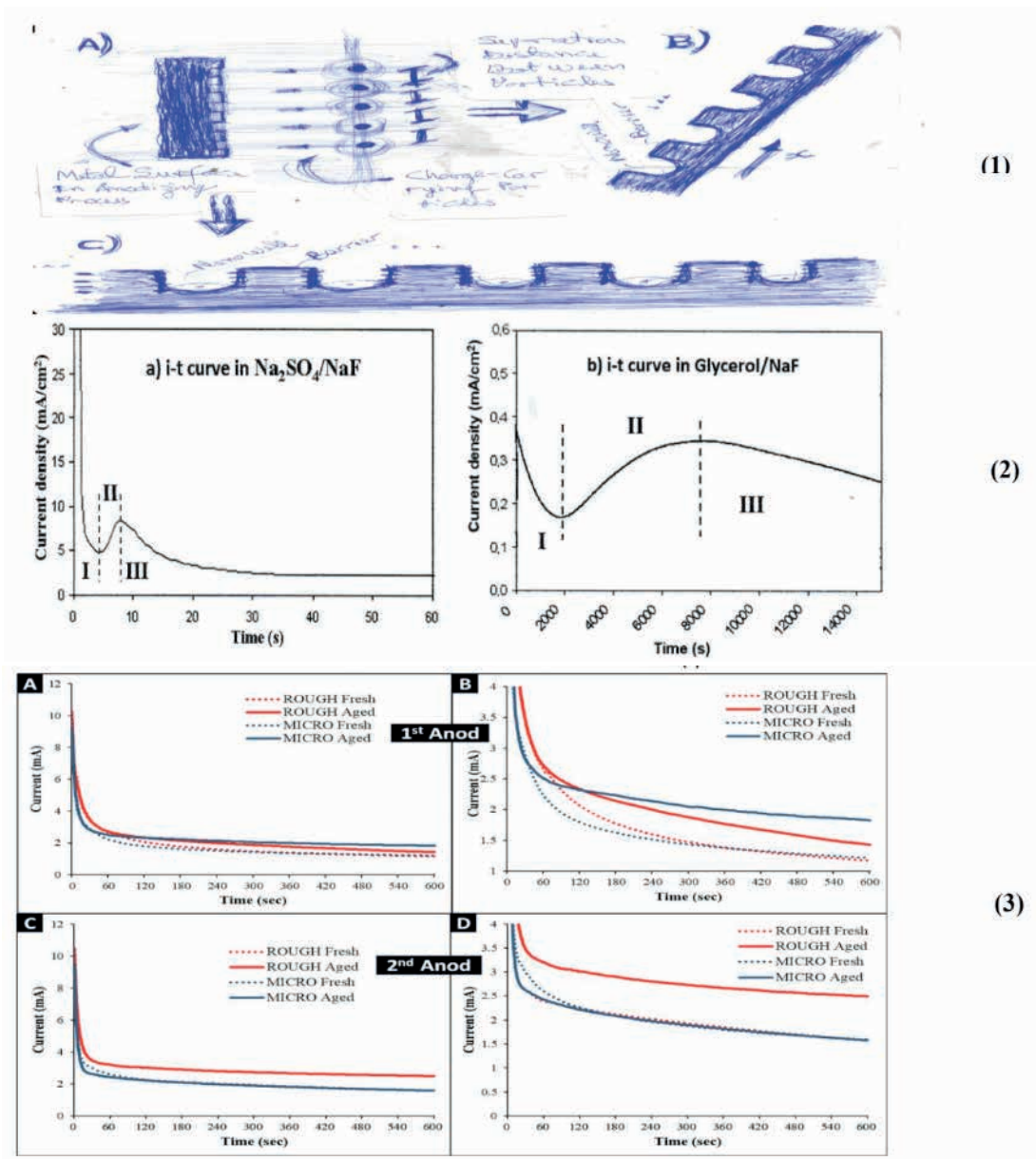
in an electrodynamic anodizing system are analyzed, discussed, and reviewed. From this review, discussion and analysis, among others, two questions that arise are: is there a relationship between charge carrier current and nanostructure and microstructure development during the anodizing procedure? If, yes; then, how to explain this relationship in an analytical causal and also exact way? For the first question, the answer is simple and exact: yes. As for the second question, there are some proposals by some authors to establish this causality between the two contained in [4], 2022, [5], 2013, [6], 2020, [7], 2005, [8] 2023, [9], 2019, etc., but they are not exact solution of the question, that is, the problem is still open. Then, in this section, this author intends to give a broader, original and improved proposal for the causes of the behaviors and nature of the current density through these layers with or without nanostructures.

GENERALIZED CAUSES OF CURRENT BEHAVIOR IN LAYER UNDER CONSTRUCTION

Here, a new and original proposal of the causes of the behaviors and nature of the current density of charge carriers is given, but without considering the presence of nanostructures on a layer, where the charge carriers traverse, it is not a necessary condition to include them in a generic treatment for all cases.

In this path, the two graphs in Part (2) of Fig. 01 reveal that, in the **phase I**, the action-reactions, by anodization, between charge-carrying particles of the electrolyte and the metal surface, or between those of either of these media, begin under an applied and constant external potential in volts, 20 V. However, notice that the current intensity decays exponentially as the anodization

4 Original and more comprehensive than the previous proposals already published in this area for new materials obtained by the anodizing procedure.

Fig. 01- Abstraction and experimental reality of an anodizing engineering generating layer, its nanostructures and current through them. *In the picture (1*): Simplified model of the interaction between the particles of the electrolyte and the metallic surface in ʻelectrodynamics' of anodization for formation of the nanopore (capacitor nanowell). In part A there is a lateral view of the metallic surface under the action, in frontal line, of the carriers of the same charge, in the electrolyte, during the anodizing. In part B a one-sided view of a nanowell successively interpolated by a barrier layer is shown after an 'electrodynamics' anodizing. Part C is another imagination of the possible one-sided images of the capacitor nanowell followed by a barrier and the barrier followed by the capacitor nanowell, successively, up to the surface edge. *In the graphics in (2*): Current density through the layer with its nanostructures growing on the titanium metal surface, when subjected to anodizing engineering. In a), anodizing took place in NaF/H_2O under 20 V. In b), anodizing took place in Glycerol/NaF and under the same voltage. (experimental source of data obtained by Regonini *et al* [5] and granted use)**.** *In the graphics in (3*): Current behavior as a function of the construction time of the anodized layer on the surface of Rough-Ti or Micro-Ti and forming nanostructures on this layer. Anodizing was carried out using new electrolyte (*fresh*) or using electrolyte already used in a first anodizing (*aged*). In A and B: results for single-step anodizing, under 60 V for 10 min each. In C and D: results for two-step anodizing, where the second anodizing was conducted under 60 V for 10 min each, after removing the anodized layer, in the first anodizing step, under 75 V, for 120 min each and with fresh electrolyte. (Here, these experimental results are of concession from the original: Li *et al* [13])

time increases, and reaches a well-defined minimum at the end of this phase. By the modern electrodynamics of Quantum Mechanics Theory, the cause of this behavior is that the layer consisting of particles (electrolyte and surface) is associated with a barrier potential, which resists the passage of charge carriers in motion (current).

Thus, during anodizing, as a function of time, the layer grows, it takes on a massive form, progressively larger, more defined, more robust and compact. These characteristics of the layer are carried over to its associated barrier potential, which causes the current intensity to be braked exponentially and as much as possible that the particles in the current do not reject compression or action: first minimum of current intensity, see graphs **a)** and **b)** in Part (2) of Fig. 01. At this moment, the charge carriers of the current are at their maximum allowable limit of compression, so, by action-reaction, they tend to establish a balance between forces with the constituent particles of the barrier (layer). But that does not occur at this moment of transition of the current's behavior. Here, it is the end of phase **I**, and the first attempt to seek a balance between forces of the particles of the barrier and the current, but that does not occur at this moment, because the layer is in its initial growth.

In this attempt to establish a equilibrium between forces, the **phase II** begins, where the current of charge carriers, at the maximum of its compression by the increasing barrier potential, reacts strongly and progressively to the maximum of the interaction it has against this potential. Then, at that point, the current ceases to be retarded and starts to increase progressively or almost abruptly through the barrier (*spring* or *wave effect*).

But, the surface layer is growing through anodization and, therefore, there is an increase in the density of charge carriers at its base, which makes the barrier potential brakes the current increase progressively or, even, abruptly. And, under voltage, as long as the equilibrium of forces between the current particles with the layer (barrier) particles is not reached, the current intensity is still being braked down, but advancing or increasing. Then, when it is close to this equilibrium or starting this equilibrium, the current intensity reaches a local maximum, which characterizes the end of the phase **II** for the current behavior. This local intensity maximum can be seen in the graphs in **a)** and **b)** in Part (2) of Fig. 01.

In the vicinity of this equilibrium, the constituent particles of the current density are exhausted in their maximum possible action against and on this layer (potential barrier). Then, the **phase III** begins,, where the layer becomes robust and resistant in its internal interactions. Thus, consequently, its associated barrier potential prevents the increase in the current flow of charge carriers, which tends to decrease to a constant flux value when this equilibrium is reached. In other words, the constant current flow indicates that an equilibrium between formation and dissociation of charge-carrying particles has occurred in the `electrodynamics' of anodization.

But, if during anodizing, the internal interactions in the electrolyte are strong, comparatively, to the action of the DC voltage, i.e., if the electrolyte resistance is intense, then it results that the internal interactions of the layer and its associated barrier potential become more intense. And, they reach their maximum reaction potential against the passage of moving charge carriers (current), when equilibrium is stabilized. Consequently, therefore, the current flow is little at the beginning of phase **III**, and, gently, starts to decrease until it becomes constant at the end of this phase, but over a long time, as is shown in the graph in **b)** in Part (2) of Fig. 01, for

example.

However, keeping the same DC voltage, if it is the other way around, i.e., if the internal interactions of the electrolyte are weak, comparatively, to the action of the DC voltage, then it results that the internal interactions of the layer and its associated barrier potential become less intense than in the previous case. And, they reach their maximum reaction potential against the passage of moving charge carriers (current), when equilibrium is reached. Consequently, in this case, there is more current flow at the beginning of phase **III**, and, in a sharp trend, it starts to decrease until it becomes constant at the end of this phase, but all in a short time, as is shown in the graph in **a)** of the same Part (2) in Fig. 01, for example.

Then, in a generalized view, these are the causes of the behavior of the current of charge carriers through a layer, which is associated with a barrier potential, building over time and under external voltage.

ANALYTICAL CAUSES OF THE RELATIONSHIP BETWEEN CURRENT AND NANOSTRUCTURES IN DEVELOPMENT

In this part, continuing the review started in the preceding subsection, a new and original causal proposal is given for the current behaviors related not only to the layer but also to its growing nanostructures when the layer is built on a metal base subjected to anodization electrodynamics. In this case, *picture* (1) and its description in Fig. 01 represents an abstraction of anodization electrodynamics for fabrication of ordered nanostructures and current through the layer under construction.

In this direction, notice the reader that in the causes of the current flow behaviors, given in the previous subsection, it was not necessary, initially, to include effects attributed by nano and microstructures. Possible and correct treatment, because the causes have their foundation in the electrodynamics of particles in motion, in their total form, where there is a potential barrier to be crossed. Electrodynamics, where it is known that the density of particles and their transmission coefficient, through a potential barrier, present oscillations in their form as a function of the incident energy of the particles. And, these oscillations start to be damped and undone as the energy of the barrier becomes robust in comparison to the energy of the particles, which makes the crossing action. As an example, such causes and explanations are reflected, again, in the behaviors of the current density of charge carriers, progressively, displayed in **phases I**, **II** and **III**, of each graph, displayed in **a)** and **b)** of Part (2) in Fig. 01 and, also, in the graphs of (mA/cm^2) x time prepared by Mohan *et al* [6], 2020 and by Mor *et al* [7], 2005 among others.

Then, the relationship between current intensity and nanostructures on a layer under construction, on the base of a metal, depends on the comparison between the energies of both: of the particles constituting the current and the layer or potential barrier. Therefore, **in the first case**, if the interaction between the internal particles of the layer is intense, comparatively, the interaction or energy of the particles constituting the current, then the presence of the nanostructures, for example: pores, on the layer under construction, is not seen (felt) by the particles of the current. In another way, it can be said that, sensitively, the moving particles, which constitute the current, are not affected by the nanostructures forming on the surface of that layer.

Consequently, in this case, the current density should decrease with a behavior of the exponential form as a function of the building time of the layer, with or without nanostructures on its surface. Such

a consequence is reflected in the current behaviors exhibited in the experimental results given in the plots in **A** through **D** of Part (3) in Fig. 01. In addition, this behavioral consequence is reflected in the current densities as a function of anodization time given in the graphs performed by Gulati *et al* [14], 2015 and, more recently, in the graphs (mA/cm^2) x anodization time performed by Guo *et al* [4], 2022: experimental results for short anodization times (of the order of 60 s). In this case, it becomes more complicated to detect the presence of nanostructures, on the surface of the layer, by the behavior of the flux or current density through that layer under construction.

However, **in the second case**, if the interaction between the internal particles of the layer is weak or about the same, comparatively, as the interaction or energy of the constituent particles of the current under DC voltage, then the presence of the nanostructures, for example: tubes, on the layer under construction, can be seen by the particles of the current. In other more exact words, sensitively, the moving particles, which constitute the current, can be affected by the presence of the nanostructures forming on the surface of that layer.

As a consequence, the behavior of the current should be oscillatory as a function of the growth time of the layer or its potential energy. But now, the presence of the nanostructures, for example: wells, contribute to make the oscillations much more defined and with well-localized maximum and minimum peaks. Thus, this cause and behavioral effects indicate that there may be a network of nanostructures forming on the surface of this layer. Therefore, through these effects, nanostructures can be detected or their formations over the layer under construction inferred. Such more refined, selective and specific consequences for current behavior

are reflected in the current densities as a function of anodization time exhibited in **phases I**, **II** and **III,** of each graph, in **a)** and **b)** in Part (2) of Fig. 01, which was analyzed in a generalized view (preceding subsection), to introduce here effects of nanostructures on current. Also, these fine-grained effects, for the current behavior, are manifested in the graphs of (mA/cm2) x anodization time published by Mohan *et al* [6], 2020, Mor *et al* [7], 2005, Yang *et al* [9], 2019, and by several other researchers in this experimental area of new materials or biomaterials.

Thus, is established a new and original cause-effect proposition of the relationship between nanostructures, on the surface of the layer being built on a metal base, and flow or current density of particles through that layer and its nanostructures.

DEVELOPMENT OF THE NANOSTRUCTURES WITH THE PHASES OF THE CORRETE BEHAVIOR

Here, by the causal proposal for the effects manifested in the behavior of the flux or current density, which was given in the previous two subsections, a more exact and comprehensive answer is given to the important question: In which phase or moment of the current (**I**, **II** or **III** in Fig. 01), can nanostructures be originated?

In this sense, the foundation of the causes for the observed effects on the current behavior is the interaction between particles of the current with particles of the layer under construction and its nanostructures. Then, the phase of the current, associated with the origin of the nanostructures, depends on the nature of the electrolyte, the anticipated preparation of the metal surface, and the external voltage on the anodizing engineering, which triggers these interactions in non-spontaneous cases.

Therefore, if the base surface undergoes

mechanical, electrochemical or other external action that produces impressions, for example: in the form of nanopores, on its surface prior to anodizing, then, by induction, nanostructures can be originated from some point in the oscillatory **phase I**, example, in Part (2) of Fig. 01, or at some early point in the decaying exponential, example, in Part (3) of the same figure, of the behavior of current versus anodizing time.

But if, before anodization, the base surface does not undergo any external action aimed at inducing nanostructures, then the phase **I** of the oscillatory behavior or the initial moments of the exponential drop of the current, as a function of time, may be associated with a thin layer under construction, without containing nanostructures on its surface, which offers a potential barrier against the passage of the particles that constitute the current. Thus, it is more likely, of the nanostructures to originate from some point in the **phase II** of the current's behavior. For example, at the beginning of the phase **II**, which corresponds to the first local minimum of the oscillations given in the curves in Part (2) of Fig. 01. Case that occurs, if the interactions between the particles forming the layer are not too intense not to be broken and do not occur generating chaos in the anodizing electrodynamics conducted under external voltage.

However, as for the increase in current intensity, in this **phase II**, only, it is not due to the presence of the nanostructures, as has been stated in the cited scientific references and in all others in this area. But, the larger fraction of this increase, mainly, may be because of the oscillations that arise in the behavior of the current through the layer, even when no nanostructures are originated on the surface of this layer under construction. A case that has already been explained in the previous two subsections of this article. Therefore, at this phase, the contribution of

nanostructures to the current increase may be small. Then, care should be taken to consider **both** contributions to form the current profile shown at this phase.

With theoretical certainty, finally, from the causes and their consequences for the behaviors of the flux or current density and nanostructures, proposed here and proven in the anodization experiments, it can be said that:

> "Approximately, at ½ (half) of the anodizing time or ½ (half) of the current flow time is when occurs the full density and defined shape of the nanostructures. The anodization time or total current flow time, here, should end far enough from the beginning of the phase III to make the current flow as stationary as possible, constant. There being only phase I, in the current behavior, the total current flow time should have its end, when the current is well stationary, constant."

CONCLUSION

The relationship between the development of nanostructures and the current of charge carriers, both generated in a layer built on a metal base, titanium, for example, by anodizing engineering, is still an unresolved and closed problem in the area of new materials (biomaterials, etc.).

A NEW CAUSALITY PROPOSAL

Then, assuming as a model, the study of `electrodynamics' of particles in the presence of an increasing barrier potential, which is associated with layer under construction on the base of a metal subjected to anodization engineering under external voltage,

a) If the interactions of the carriers of the layer, or energy of the charge carriers, comparatively, are much more intense than the interactions of the constituent particles of the current, then it results that the current intensity will decrease with an exponential behavior until it reaches a stationary

(constant) behavior and, appreciably, will not suffer effects generated by the presence of nanostructures on the layer. Therefore, in this case, detecting nanostructures by the direct behavior of the current density becomes more complicated;

b) However, **if** the interactions of the charge carriers of the layer, comparatively, are very close or smaller than the interactions, as a whole, of the constituent particles of the current, then it results that the current intensity, in besides decreasing exponentially, will present an oscillatory behavior and will suffer effects produced by the presence of the nanostructures originated on the layer, which is under construction. Therefore, each increase in current, in its phases of behavior (**phase II**, in Part (2) of Fig. 01, for example), should not be due, only, to the presence of the nanostructures. The increase in current is more because of the maximum interaction capacity, energy, offered by the constituent particles of the current, which, independent of the presence of the nanostructures forming on this layer, in this case will present an oscillatory nature. Hence, there will be **two** contributions to the behavior of the current, in this case: one originating from the barrier and the other originating from the nanostructures.

Thus, a relationship is established between current density behavior and nanostructures, where fine effects in current behavior are produced by the origin of nanostructures on the layer being built up on the surface of a metal. Then, the current density behavior, as a function of time or the energy of the layer under construction, can indicate that nanostructures are being originated and developed on the surface of the layer. Nanostructures that can originate in the **phase I** or **phase II** of the flow or current density behavior through the layer under construction (more details are in sections 3.2 and 3.3 of this paper and Fig. 01).

The starting point of this paper was from a review, therefore, the experimental graphs were taken from the scientific literature published prior to this study. But, the theoretical analyses and the proposed cause and consequences for the current and nanostructures are originally improved, more comprehensive and more accurate for the treatment of experimental results in this area. More details of this interaction (relationship) forming symmetric nanostructures are in an interesting study given in [15], 2023.

STATEMENT OF CONFLICT OF INTEREST

Also, this author declares that there are not potential conflicts of interest in the authorship and participation of the scientific work studied and prepared here for submission to institutions of foreign or national scientific publications. Grateful!

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