

Plasma dynamic synthesis of iron oxides in the «frequency operation» mode of coaxial magnetoplasma accelerator

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Abstract. It is known that the epsilon phase of iron oxide $\varepsilon\text{-Fe}_2\text{O}_3$ has the highest value of coercive force among all known simple metal oxides (~ 24 kOe) and is characterized by ferromagnetic resonance in the frequency range from 100 to 200 GHz. The noted features determine the possibility of its wide application. However, there is the problem of synthesis and stabilization this crystal structure at room temperature. In this paper, we consider the possibility to obtain $\varepsilon\text{-Fe}_2\text{O}_3$ by the plasma dynamic method when the system is operating in the frequency (multi-pulse) regime. The influence of the number power supply impulses on the phase composition of the synthesized products was studied using X-ray diffractometry.

1. Introduction

In the "iron-oxygen" system, there are 7 known phases of iron oxides such as $\alpha\text{-Fe}_2\text{O}_3$ (hematite), $\beta\text{-Fe}_2\text{O}_3$, $\gamma\text{-Fe}_2\text{O}_3$ (maghemite), $\varepsilon\text{-Fe}_2\text{O}_3$, $\zeta\text{-Fe}_2\text{O}_3$, FeO and Fe_3O_4 (magnetite), which have different structural and physical properties [1-3]. Among them, $\varepsilon\text{-Fe}_2\text{O}_3$ phase is of the greatest practical interest due to its unique magnetic properties. For example, $\varepsilon\text{-Fe}_2\text{O}_3$ phase has the highest coercivity among all known simple metal oxides (~ 24 kOe) [4]. Also it has the ferromagnetic resonance at the frequency of ~ 190 GHz [5, 6]. Such a set of properties can be useful in creating on their basis modern permanent magnets used to store information for manufacturing radio-absorbing coatings that can be used in civil and military applications. Also, the iron oxide particles, including $\varepsilon\text{-Fe}_2\text{O}_3$, are non-toxic and have a high resistance to corrosion. Nonetheless, the production of epsilon phase is a difficult scientific task, because it can exist only in a nanoscale state [7, 8] and thermodynamically unstable [9].

It is known that the plasma dynamic synthesis (PDS) method allows obtaining an epsilon phase of iron oxide and is characterized by a short reaction time (less than 1 ms), low energy consumption, high energy parameters of the synthesis process (up to 100 kJ) and high cooling rate ($\sim 10^8$ K/s). The main system element is a coaxial magnetoplasma accelerator (CMPA) with iron electrodes. Earlier it was shown that with the use of this system it is possible to achieve a preferential yield of epsilon phase [10]. In order to increase the $\varepsilon\text{-Fe}_2\text{O}_3$ phase yield in the system, it is necessary to increase the energy supplied to the accelerator from the capacitive energy storage (CES). This leads to increasing electrodynamic effects during the increased arc discharge current flowing through the main nodes of the system that can cause their destruction. To prevent possible emergencies, it is necessary to decrease the flowing current value. In this regard, in this paper it was proposed to implement the «frequency operation» mode of the system that allows reducing the basic energy parameters as well as



to estimate the influence of the consecutive power pulses number on the phase composition of the synthesized samples.

2. Experimental part

The PDS system and its main elements are shown in figure 1. The working principles and were discussed previously in details [10]. In this system, the «frequency operation» means the possibility to implement the PDS process in a series of consecutive plasma shots, occurring one after another. Shots happen behind each other for some time delay (t_{del}), which is necessary to ensure that power keys (ignitrons "CD") of the capacitors battery (C), which worked first, managed to close. Such a "frequency" mode can be realized in the system by separately feeding pulses from the pulse generator to the corresponding sections of the control blocks (CB).

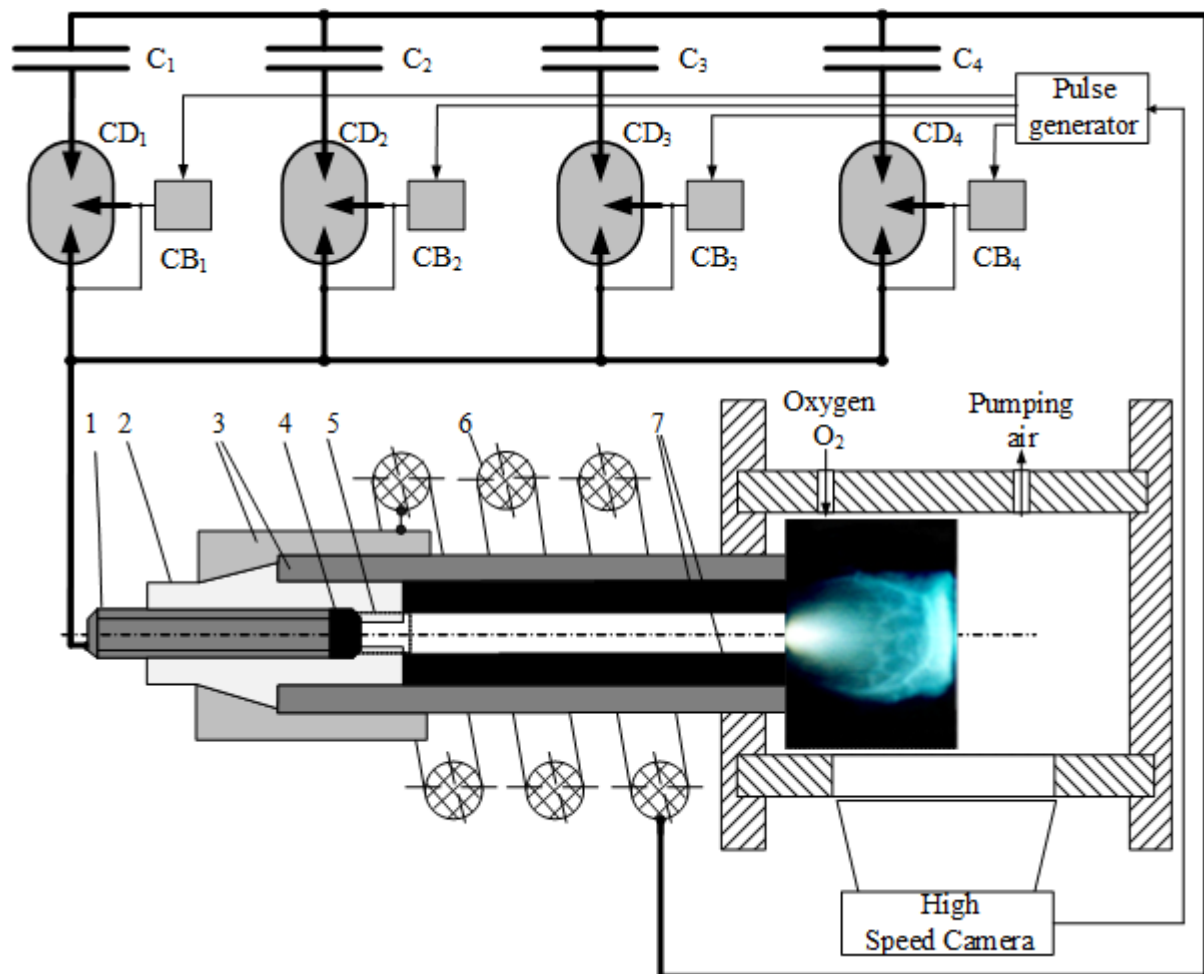


Figure 1. The system of plasma dynamic synthesis. 1 – Central electrode, 2 – Insulator, 3 – Metal hull, 4 – Iron tip, 5 – Plasma formation zone, 6 – Inductor, 7 – Iron accelerator channel.

The number of capacitive sections (C1, C2, C3, C4) included in the work was chosen in such a way that their electrical capacity was 7.2 mF each (a quarter of the total electrical capacity of the CES). Power keys (CD) were closed when delayed pulses from the generator were applied to the corresponding control blocks (CB). Thus, the sequential start of CES sections was carried out. These sections were discharged to the load. In our case, it was CMPA. In order to study the influence of number of pulses, the following possible cases were investigated:

- 2 consecutive power supply pulses ($C1=7.2$ mF, $C2=7.2$ mF);
- 3 consecutive power supply pulses ($C1=7.2$ mF, $C2=7.2$ mF, $C3=7.2$ mF);
- 4 consecutive power supply pulses ($C1=7.2$ mF, $C2=7.2$ mF, $C3=7.2$ mF, $C4=7.2$ mF).

In all cases, the charging voltage (U_{ch}) of CES was constant with the value of 3.0 kV. The initial energy parameters were selected in such a way in order to decrease approximately twice the initial accumulated energy (W_{ch}) and decrease the supplied energy (W), respectively, in comparison with the experiments shown in [10]. The energy parameters were recorded by using the digital oscillographs Tektronix TDS 2012.

The implementing every of the considered experiments resulted in obtaining the powdered products. The phase composition of these powders was studied with the X-ray diffractometry method by using a Shimadzu XRD-7000 X-ray diffractometer. The quantitative analysis of phase composition was made by using the software “Powder Cell 2.4” and PDF4 database.

3. Results and discussion

The main goals of the proposed experiments series were to reduce the electrodynamic forces at the main structural elements by decreasing the value of the flowing arc discharge current, as well as to achieve the maximum epsilon phase yield. table 1 presents the initial and calculated data of experimental series with using the different number of consecutive pulses from 1 to 4. The experiment with one operating pulse was carried out in order to show the decrease in the energy parameters in comparison with the “normal” mode of CMPA work ($U_{ch}=3,0$ kV; $C=14,4$ mF).

Table 1. The initial and calculated data of the experimental series.

N, pulses	Initial date			Calculated data						
	U_{ch}	C	W_{ch}	W	Δm	m_{pow}	Phase composition, % mass.			
	kV	mF	kJ	kJ	g	g	ϵ -Fe ₂ O ₃	Fe ₃ O ₄	α -Fe ₂ O ₃	
1	3,0	7,2	32,4	19,7	0	0	-	-	-	
2	3,0	7,2	64,8	39,9	0,4	0,23	35,0	50,0	15,0	
3	3,0	7,2	97,2	71,3	8,2	2,77	62,0	13,1	24,9	
4	3,0	7,2	129,6	73,6	12,8	4,30	64,7	8,0	27,3	

Carrying out an experiment with one operating pulse made it possible to establish a decrease in the maximum value of the current flowing in the circuit up to ~145 kA, according to the registered oscillograms of the working processes (figure 2). This value is lower than that obtained in the "normal" operating mode of the accelerator [10] (approximately at 20%). In the case of one pulse mode, the observed decrease in the maximum current value affected the values of the average discharge power (63.5 MW) and the supplied energy (19.7 kJ). This results in sufficient decreasing the mass of the eroded material (Δm) and the mass of final powdered product (m_{pow}). They were too small to carry out the analytical studies of the phase composition. This result showed the inexpediency of using the system at such low energy parameters in one pulse mode.

However, even with the implementation of the two consecutive plasma shots, both the electric erosion from the walls of the accelerating channel and the mass of the final powder significantly increased. This is most likely due to the fact that during the movement of the first plasma shot along the walls of the accelerating channel, the material from the walls did not have enough time to effectively crystallized. It leads to creating the natural irregularities on the surface. Thus, the energy released from the second shot heated the accelerating channel surface easier and more material was involved in plasma flow movement. In the cases of 3 and 4 pulses, the movement of the consecutive shots resulted in more effective sublimation of material from the walls of accelerating channel. It is clearly seen from the data about electric erosion (Δm) in the table 1. With increasing the number of pulses, both the erosion and the mass of powdered material increased.

The study of the phase composition for products synthesized with 2 and more consecutive shots confirmed the effectiveness of using the system working in the frequency operation mode. More over, according to phase composition analysis (figure 3), increase in the number of plasma shots leads to increasing the yield of epsilon phase. For example, in case of two pulse mode, the dominant phase was magnetite (50% mass), while the $\epsilon\text{-Fe}_2\text{O}_3$ content was 35.0%. With an increase in the number of working pulses, the yield of epsilon phase was increased approximately twice up to ~65 % for four pulse mode.

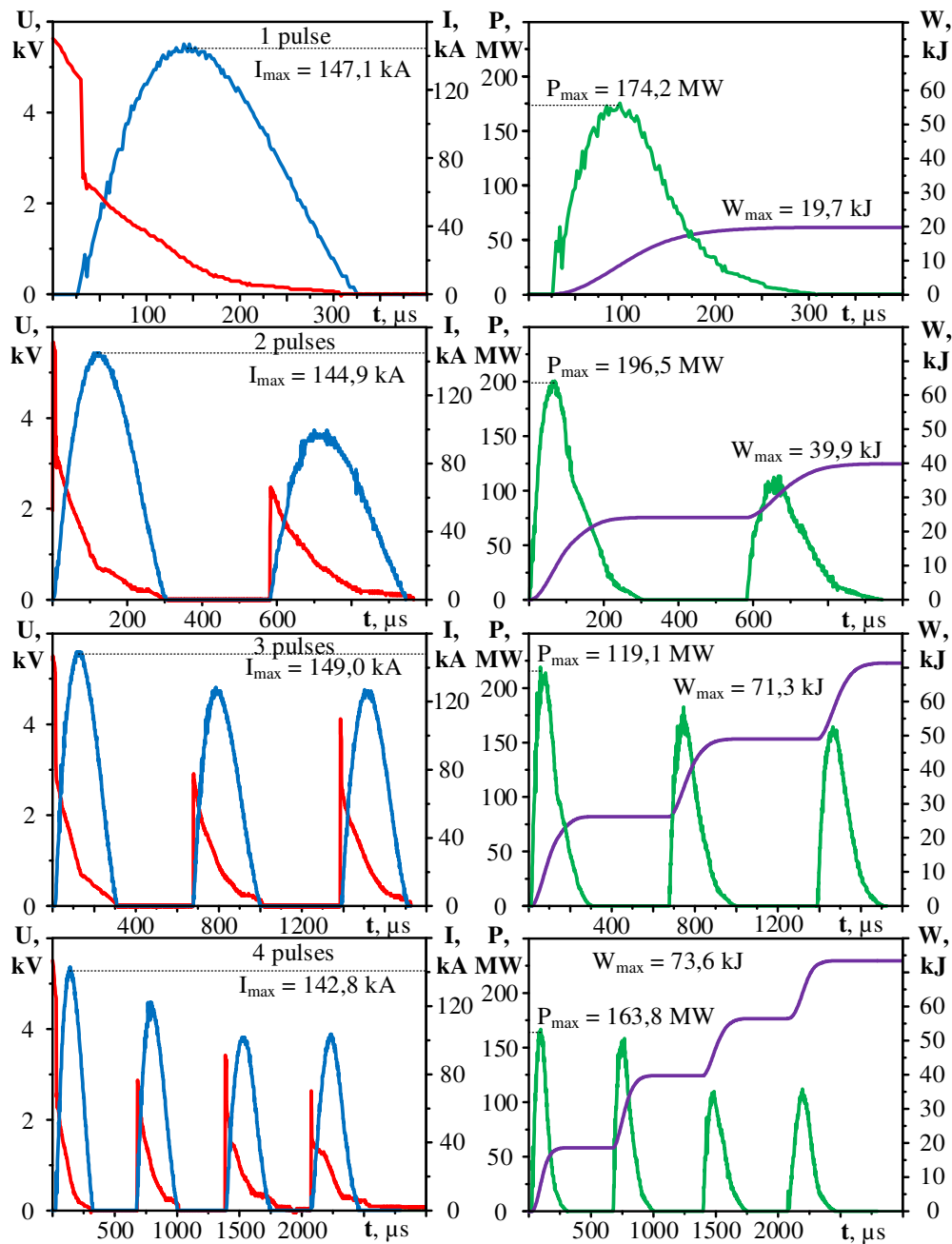


Figure 2. Working voltage oscillograms on the electrodes of the CMPA $U(t)$, the arc discharge current $I(t)$, as well as the power curves of the discharge $P(t)$ and the supplied energy $W(t)$ during the studies of the CMPA "frequency operation" mode.

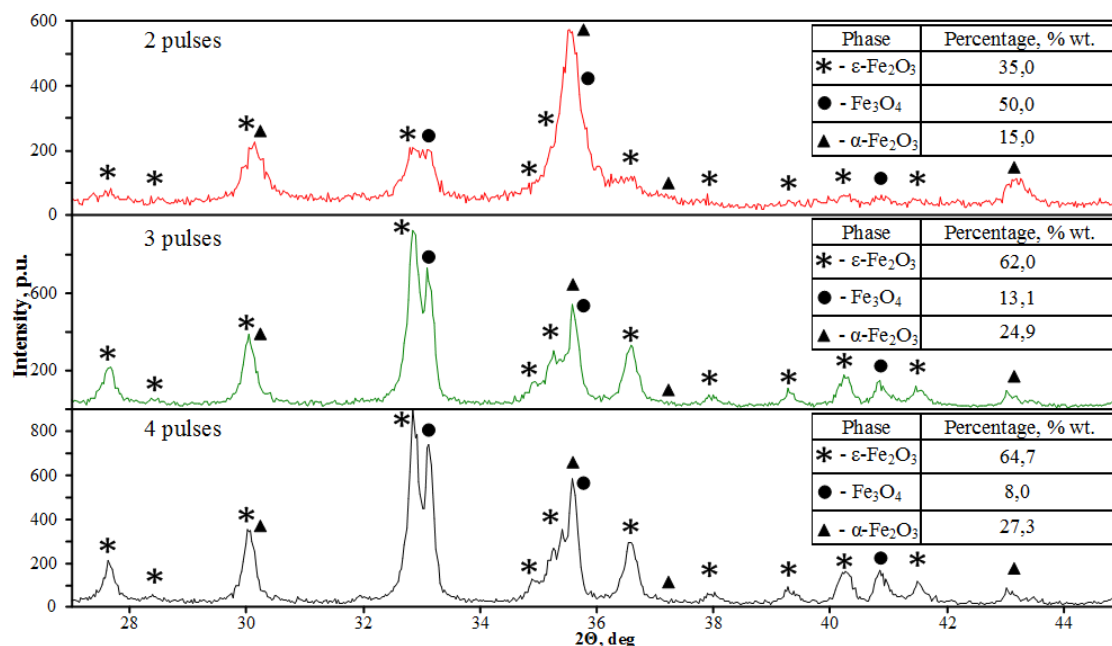


Figure 3. X-ray PDS diffractograms obtained during the CMPA "frequency operation" mode.

4. Conclusion

The obtained results allow making a conclusion about the prospects of using the "frequency" operating mode of the CMPA with a sequential working pulses. In addition to the fact that this method can be implemented at low energy parameters of the system, that has a positive effect on the system resistance to dynamic effects, it allows achieving an acceptable result from the position of ϵ -Fe₂O₃ yield (up to ~65.0%). In addition, the increase in the number of pulses was accompanied by an increase in both the eroded mass from the accelerating channel, as well as the final product mass.

Acknowledgments

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