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Study of the effect of non-thermal plasma on the structural properties of pure Cadmium Oxide thin Films (CdO) prepared by Pulsed laser Deposition Technique (PLD)

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ABSTRACT:

In this research, the preparation of pure (CdO) films on a glass substrate was conducted using pulsed laser deposition method (PLD) .The deposition of the films was done at different times (5 , 10 , 15 , 20) minutes at room temperature . The effect of non-thermal plasma on these prepared films in two exposure times of (30, 60) minutes was studied. The results of X-ray diffraction (XRD) of the prepared pure (CdO) films showed that the films have polycrystalline structure of cubic phase with preferred orientation (111) . It was noted that the increasing of deposition time of (Nd : YAG) laser , reduces the intensity of diffraction peaks at (111) , (200) , (220) . The results of (XRD) have also showed that when the prepared pure (CdO) films are exposed to non-thermal plasma , their crystalline structure was not affected . The tests also showed that the increasing of the exposure time to non-thermal plasma causes a decrease in the intensity of the diffraction peaks. The (AFM) tests also demonstrated that the changing in (RMS) of the surface roughness is irregular and it was found that the (RMS) increases by increasing the deposition time.

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دراسة تأثير البلازما الباردة على الخواص التركيبية لأغشية اوكسيد الكادميوم النقية المحضرة بتقنية الترسيب بالليزر النبضي

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الكلمات المفتاحية:

أوكسيد الكادميوم النقي
الترسيب بالليزر النبضي
البلازما الباردة
الخواص التركيبية

الـــخُـــلاصـــة:

في هذه الدراسة تم تحضير ودراسة الخصائص التركيبية لأغشية اوكسيد الكادميوم النقية على قواعد زجاجية والمحضرة بتقنية الترسيب بالليزر النبضي (PLD) وبأزمان ترسيب مختلفة (20,15,10,5) دقيقة , وبدرجة حرارة الغرفة وتأثير البلازما الباردة على هذه الأغشية المحضرة بحالتين للتعرض (60,30) دقيقة , واظهرت نتائج حيود الأشعة السينية (XRD) لأغشية (CdO) النقية المحضرة أن تركيب الأغشية كان متعدد التبلور (Polycrystalline) من النوع المكعب (Cubic) , وأن الاتجاه المفضل للنمو هو (111) للأغشية المحضرة قبل وبعد التعرض للبلازما الباردة , وتبين ان بزيادة زمن الترسيب بالليزر نيدميوم – ياك (Nd:YAG) قلل من شدة قمم الحيود بشكل قليل جداً للذروات (111), (200), (220) , كما تبين من نتائج (XRD) عند تعرض أغشية (CdO) النقية المرسبة للبلازما الباردة لا تؤثر على طبيعة التركيب البلوري , وتبين ان بزيادة زمن التعرض للبلازما الباردة ادى الى انخفاض في شدة قمم الحيود بشكل قليل , واظهرت نتائج فحوصات (AFM) ان التغير في (RMS) غير منتظم الا انه بزيادة زمن التعرض للبلازما الباردة يصبح التغير

في (RMS)الى حد ما منتظم ونلاحظ زيادة (RMS) بزيادة زمن الترسيب.

1. INTRODUCTION

Cadmium oxide thin film is considered as a semiconductor of (n-type) , it is found in nature in two structural forms , the first one is the crystalline form and the second is the amorphous form , the crystalline form is of reddish brown colour , while the amorphous form has no colour [1] . The crystalline structure of pure cadmium oxide is face centered cubic (fcc) [2] . The pure cadmium oxide is reddish brown while the impure cadmium oxide has different colours because of the defects found in its structure as a result of the ionic vacancies [3] . The cadmium oxide (CdO) is used as thin films in the manufacture of liquid crystal display (LCD) , IR-detectors and the thin films transistors [4] . The C.S. of CdO is cubic with fcc phase fig (1) . In this research we prepared thin films of CdO by PLD with different deposition time and studied the effect of time exposure of non-thermal plasma on these films. Non-thermal plasma is also called low temperature plasma , this type of plasma has partially ionized gas particles and has kinetic energy (1eV) . The non-thermal plasma is the type of plasma that is used in most of the scientific researches . It is classified into two kinds : equilibrium plasma and non-equilibrium plasma [5] .It has been widely used in industry especially in preparing and depositing thin films [6].

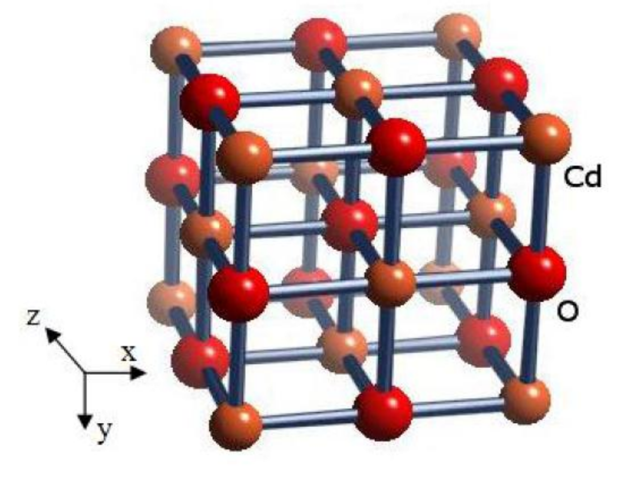

Figure 1: the crystalline structure of (CdO) [7].

2. EXPERIMENTAL WORK

The pure (CdO) powder has been used as a material supplied by (Aldrich Chemical Company , Inc) from Germany and the purity was (99.9%) after the (XRD) test has been made , then the powder of pure cadmium oxide (CdO) has been compressed to make pellets using hydraulic compressor manufactured in china with compressing power (8 tons) , the pellets' diameter was (2 cm) and its thickness was (3mm) , rectangular glass substrates have been used of dimensions (7.6 x 2.5) cm^2 and their thickness (1mm) made of borosilicate glass supplied by (China National Machinery) to deposit the samples on them , the glass substrates have been cleaned before they were used in the deposition process .Thin films were prepared through depositing pure (CdO) using (PLD) method on glass substrates made for preparing thin films , steps have been followed to prepare films homogeneously like cleaning the deposition system well using highly pure alcohol or acetone to make it ready for deposition .Then the glass substrate was placed on substrate holder in front of the target surface so the target surface becomes parallel to the base substrate permanently , then the target surface is placed on a rotary target holder , the distance between the glass substrate fixed on the holder and the target surface was (5.5 cm) and it is fixed for all the films , then the laser beam (Nd : YAG) , with laser energy (100mJ) wavelength (1064 nm) frequency (5Hz) pressure (0.5×10^{-1} mbar) , at room temperature and through the vacuum chamber , has been focused on and the rotary target surface inside the deposition chamber and the angle was 45^o and then all the valves of this system were closed as well as the vacuum chamber , then the system was emptied from the air to get pressure of (0.5×10^{-1} mbar) inside the deposition chamber through using rotary pump , the deposition of the films was made at different times (5 , 10 , 15 , 20) minutes .The vacuum chamber made of Bayrex glass . It's dimensions are , diameter (20cm) , height (30cm) , thickness (5mm) , it has closed top , open bottom and cylindrical form . The thickness of prepared pure (CdO) thin films has been measured using interference photic method and (He-Ne) of wavelength (632.8 nm) and an angle (45^o) ,and the thickness was measured through the following formula [8]

$$t = \frac{\Delta\times}{\times} \cdot \frac{\lambda}{2} \quad \ldots\ldots. (1)$$

Where : t : is the film thickness (nm). ×: is the shining fringe width (nm) . Δ× : is the opaque fringe width (nm) . λ : is the wavelength of incident laser beam (nm) . The interplanar distance was calculated (d_{hkl}) between the reflective and identical surfaces to the angles their values are mentioned above from the formula [9].

$$n\lambda = 2d_{hkl} \sin \Theta \ldots\ldots\ldots\ldots(2)$$

Where : d_{hkl} : levels interplanar distance (hkl) , Θ : Bragg's angle , λ : X-ray wavelength n : interference rank . The average grain size of pure (CdO) films prepared by Scherr's formula has been calculated [10].

$$D_{ave} = \frac{0.9\lambda}{\beta cos\theta} \ldots\ldots\ldots\ldots (3)$$

Where : D_{ave} : is the average grain size , β : (FWHM) (full-width at half –maximum.

2.2 The exposure of deposited (CdO) to non-thermal plasma

The plasma system was prepared and the electrodes (Cathode and Anode) were cleaned well .These electrodes are made of Aluminum the distance between the electrodes was (4cm) , it was constant for all the samples ($5x10^{-1}$ mbar) pressure was gained , and after the samples were exposed to non-thermal plasma at different times (30 , 60) min . (I = 14mA , V= 375 V) constant for samples.

3. RESULTS AND DISCUSSION

3.1 The study of the structural properties of pure (CdO) thin films.

3.1.1 X-ray Diffraction (XRD)

The results of X-ray diffraction (XRD) of the prepared pure (CdO) films using (PLD) method (Nd : YAG) , showed that the films have polycrystalline structure and of cubic phase . At different deposition times (5 , 10 , 15 , 20) minutes . Figure (2) showed the emerging of several peaks in the diffraction angles [2Θ =33.01 , 38.31 , 55.3] degree , that is similar to the [hkl = (111) , (200) , (220)] planes respectively , before the exposure to plasma . And the crystalline level , that has distinguished direction , was (111) for all the prepared films .When comparing these results with the results of the previous studies and researches it was found that they agree to a great extent with the results of researchers [11,12] . Figure (2) shows a comparison between deposition times (5 , 10 , 15) min , and it was observed that the increase of deposition time using (Nd:YAG) laser , decreases the intensity of the diffraction peaks at (111) , (200) , (220) , and deposition time at (15) min seems to have lower peaks than the two other times (5 , 10) min , and when comparing this result to the results by the researchers , it was observed that it agrees with [13].When comparing the results gained from the diffraction angles (2 Θ) , facing the distinguished peaks positions of the prepared films samples and the surface interplanar (d_{hkl}) , to values of the American card (ASTM) numbered (00-005-0640) it was found that the results were identical to some extent before and after the exposure to non-thermal plasma , as shown in table (1) . The results of (XRD) showed that the exposure of the prepared films , using (PLD) method , to non-thermal plasma caused the increase of the films' growth , and that is clear when the exposure time to non-thermal plasma increases (60 min) , especially at deposition time (20 min) . The exposure time of the films that have higher thickness led to the regulating and ordering of the prepared films well . And so the intensity of the surface reflections (111) , (200) , (220) and (311) became higher as shown in the figure (3) . It was observed that the average grain size increases by the increase of the thickness before the exposure to plasma the reason for that is the decrease of (FWHM) according to formula (3) that means the thickness increase led to the increase of crystallization and the decrease of crystal defects . When comparing this result to the results by the researcher , it was observed that this result agrees with the researcher's results [14] . After the exposure to plasma we notice that the average grain size decreased by the increase of the deposition time . The (FWHM) has also been calculated before and after the exposure to plasma , and it was found that the value of (FWHM) decreases by the increase of deposition time before the exposure to plasma , so the average grain size increases by the decrease of (FWHM) according to formula (3) as shown in the table (1) . The results of (XRD) showed that when the deposited films exposed to plasma we see that the non-thermal plasma has no effect on the crystalline structure , the test showed that the polycrystalline structure of the films remain the same and at the same preferred direction (111) .The (XRD) test also showed that the increase of exposure time to non-thermal plasma caused the decrease of the intensity of the diffraction peaks as shown in figure (3). Table (1) explains these results and when these results are compared to the ones by the researcher , it was found that these results agree with the researcher's results [15].

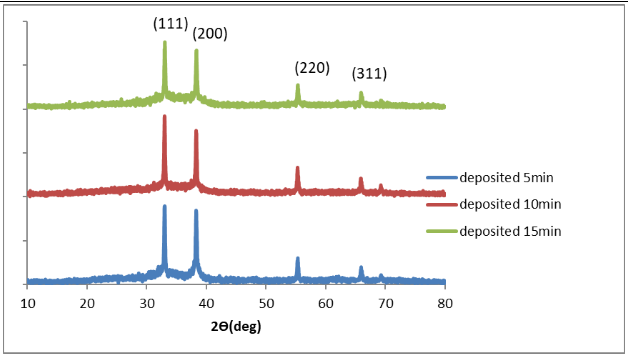

Figure 2: shows (XRD) pattern of pure (CdO) films comparing the deposition time before the exposure to non-thermal plasma.

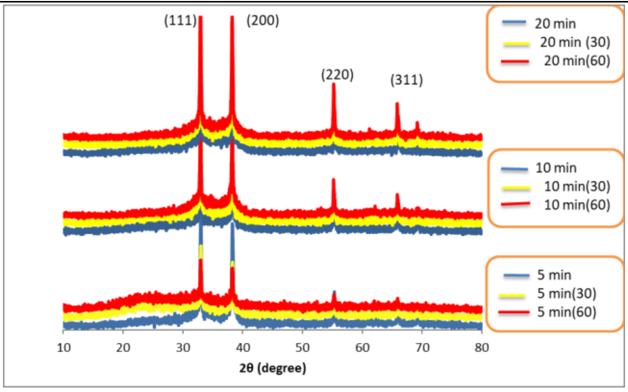

Figure 3: shows (XRD) pattern of pure (CdO) films at deposition times (5,10,20) min before and after the exposure to non-thermal plasma.

Table 1: shows the results gained from the (XRD) patterns of pure (CdO) films.

Sample No.	State	Time Deposition (CdO)	2Θ Degree	d (Å) observed	FWHM Degree	(hkl)
1	Before the exposure to plasma	5 min	33.01	2.71	0.207	111
			38.31	2.34	0.23	200
			55.3	1.65	0.22	220
			65.94	1.41	0.21	311
2		10 min	33.01	2.71	0.19	111
			38.3	2.34	0.2	200
			55.29	1.65	0.19	220
			65.92	1.41	0.18	311
3		20 min	33.05	2.7	0.21	111
			38.33	2.34	0.22	200
			55.33	1.65	0.21	220
			65.94	1.41	0.2	311
4		5 min	33.03	2.7	0.15	111

	After the exposure to plasma 30 min		38.31	2.34	0.18	200
			55.3	1.65	0.17	220
			65.92	1.43	0.17	311
5		10 min	32.98	2.71	0.18	111
			38.28	2.34	0.198	200
			55.27	1.66	0.188	220
			65.91	1.41	0.193	311
6		20 min	32.96	2.71	0.18	111
			38.26	2.35	0.19	200
			55.24	1.66	0.17	220
			65.87	1.41	0.17	311
7	After the exposure to plasma 60 min	5 min	33	2.71	0.18	111
			38.3	2.34	0.23	200
			55.28	1.66	0.24	220
			65.88	1.41	0.2	311
8		10 min	32.96	2.71	0.2	111
			38.24	2.35	0.2	200
			55.23	1.66	0.19	220
			65.9	1.41	0.18	311
9		20 min	32.96	2.71	0.17	111
			38.25	2.35	0.18	200
			55.24	1.66	0.17	220
			65.87	1.41	0.18	311

Table 2: shows some structural parameters of pure (CdO) films.

Sample No.	state	time deposition (CdO)	(hkl)	2Ɵ degree	Average crystalline Size (nm)	a (Å)
1	Before the exposure to plasma	5min	111	33.01	41.81	4.69
2		10min	111	33.01	45.55	4.69
3		20min	111	33.05	41.22	4.68
4	After the exposure to plasma 30 min	5min	111	33.03	57.7	4.68
5		10min	111	32.98	48.08	4.69
6		20min	111	32.96	48.07	4.69
7	After the exposure to plasma 60 min	5min	111	33	48.08	4.69
8		10min	111	32.96	43.91	4.7
9		20min	111	32.96	50.9	4.69

3.1.2 Investigating by Atomic Force Microscopy (AFM)

The (AFM) has been used to study the structural properties of the pure (CdO) films prepared by (PLD) method before and after the exposure to non-thermal plasma into two dimensions (2D) and three dimensions (3D) .The results of (AFM) of the thin films surfaces of pure (CdO) material as shown in figure (4) , which shows that the films prepared are in general homogenous and have no vacancies . It was observed that the surface roughness increases by the increase of the plasma deposition time and it is a normal result because the increase of the thickness leads to the increase of the films' growth, so figure (4) may agree with our explanation mentioned above . On the other hand, the researches [12 , 16] , agree to a great extent regarding the increase of the films thickness and plasma deposition time may lead to this result . So the results of the surface roughness and (RMS) , as shown in figure (4) , showed that the changing of (RMS) is not constant , as shown in the table (3) , however it is possible that the increase of exposure time to non-thermal plasma will make the change of the (RMS) be constant to some extent , and it was observed the increase of the (RMS) by the increase of deposition time so it is possible that the increase of plasma exposing time caused the increase of the growth of crystalline grains , as shown in the figures , especially the (3D).

Table 3: shows the values of (RMS), the roughness (S_a) of pure (CdO) films before and after the exposing to non-thermal plasma.

State		Time (min)	Roughness (S_a) nm	RMS nm
Before Exposure to Plasma		5	375.1	472.76
		10	693.54	912.31
		20	326.34	407.44
After Exposure to Plasma	30 min	5	376.07	479.59
		10	292.34	378.06
		20	909.71	1119.9
	60 min	5	170.48	234.27
		10	319.48	409.64
		20	431.29	601.27

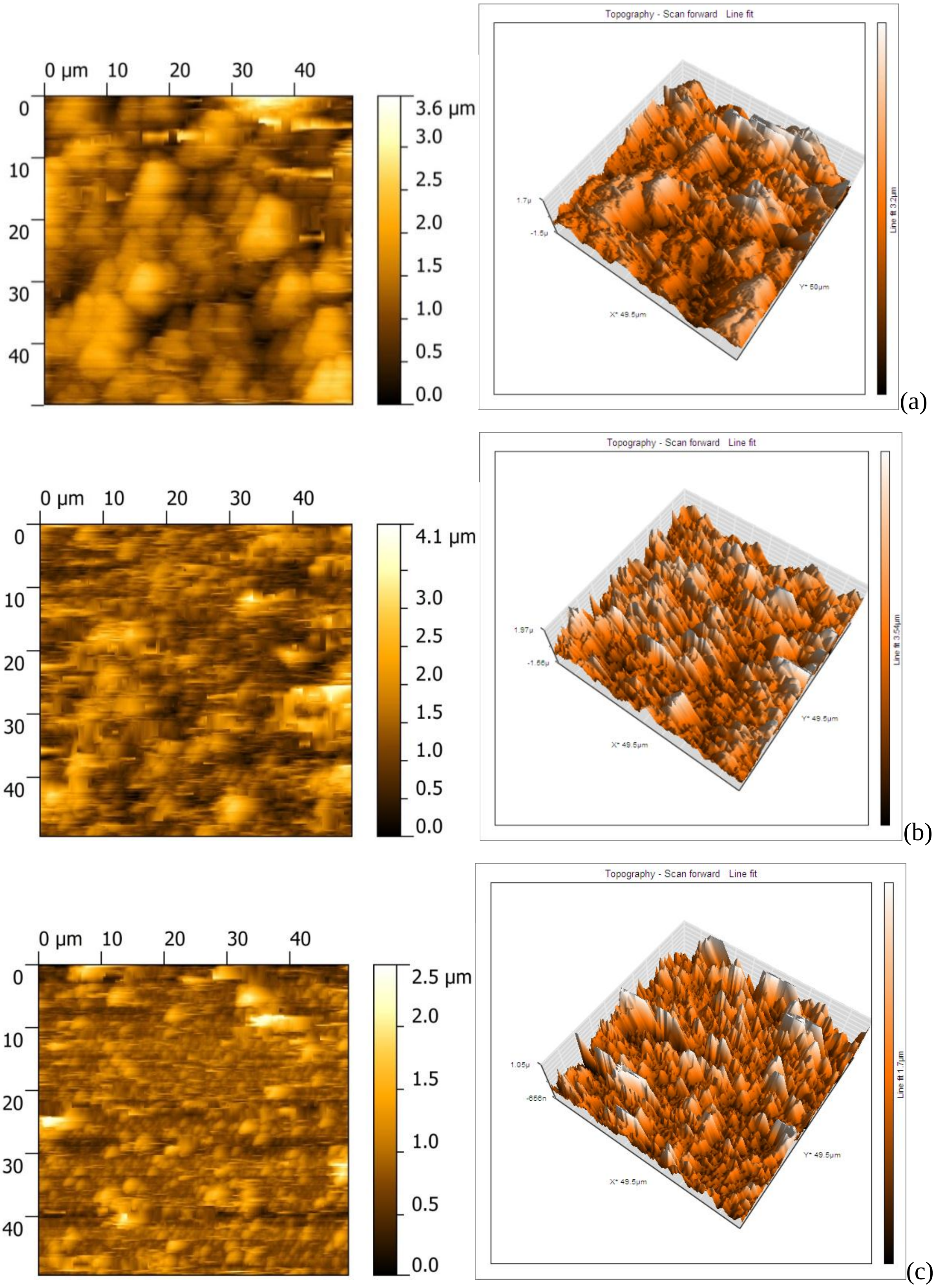

Figure 4: shows the results of (AFM):

a) **Before exposure to plasma (for 5 min deposition)**
b) **After exposure to plasma (30 min) (for 5 min deposition)**
c) **After exposure to plasma (60 min) (for 5 min deposition)**

4. CONCLUSIONS

After studying and discussing the results obtained from this research , we reached to the following conclusions.

1- The (XRD) of pure (CdO) films prepared by (PLD) method at room temperature showed that they have a polycrystalline structure and of a cubic type .

2- The (XRD) showed that the increase of the deposition time using (Nd :YAG) laser decreased the intensity of the diffraction peaks , it has also showed that the increase of deposition time to non-thermal plasma led to an increase of the intensity of the diffraction peaks . The results of (XRD) have also showed that when the prepared pure (CdO) films are exposed to non-thermal plasma , their crystalline structure is not affected .

3- (AFM) tests showed that the films prepared are in general homogenous and have no vacancies . So the results of the surface roughness and (RMS) showed that the changing of (RMS) is not constant , however it is possible that the increase of exposure time to non-thermal plasma will make the change of the (RMS) be constant to some extent .

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