

Study of the structural and morphological characteristics of the $Cd_xTe_yO_z$ nanocomposite obtained on the surface of the CdS/ ZnO heterostructure by the SILAR method

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Abstract

 $Cd_xTe_yO_z$ nanocomposite films were grown by the SILAR method on the CdS/ZnO surface during cyclic processing in precursor solutions followed by removal of excess reagent from the surface of the substrate by washing in hydrogen peroxide. To stabilise the surface states and saturate with oxygen, the surface was annealed in a diffusion furnace. XRD, RAMAN, and SEM analyses were used to analyse the phase composition, structural, and substructural parameters. The nanocomposite film consists of different types of oxides, namely trigonal TeO₃, Monoclinic TeO₄ and CdTe₃O₈, orthorhombic TeO₂ and CdTeO₃. The formation of films is explained using the Volmer–Weber growth mechanism. SEM analysis of the formed nanocomposite showed the presence of nanometer-scale globules. Partial amorphization of the heterostructure occurs due to the presence of nanometer-sized particles.

Keywords SILAR method · Cadmium-tellurium oxides · Electrolyte · Nanocomposite · Heterostructure

1 Introduction

In recent decades, researchers have focused on the acquisition of thin semiconductor films due to the breadth of their application in various fields of optoelectronic technology [1–7]. Thin films based on materials from the A^2B^6 group deserve special attention due to the possibility of applications in solar energy, lasers and sensors [3, 4, 8, 9]. Thus, cadmium semiconductors, in particular CdTe, CdSe, CdS, etc., are widely used [5, 6, 10, 11]. CdSe is considered promising for the creation of thin film transistors and gammaray detectors due to the direct band gap and the ability to

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adjust resistivity in wide ranges [7, 12]. In addition, studies of CdTe are of great interest, which is rightfully considered a semiconductor of technological importance due to its wide use in solar energy [8, 9, 13-19].

Not less promising and interesting are oxide semiconductors and heterostructures or semiconductors with a layer of their own oxide on the surface [20, 21]. Synthesised arrays of ZnO nanowires demonstrated excellent homogeneity in morphology and geometric characteristics, which may be beneficial for efficient electron transport [22]. At the same time, the main drawback of this material is the wide band gap, which limits the absorption and use of the visible region. To optimise functional properties and increase photocatalytic activity, narrow-band semiconductors such as CdS, CdSe, and CdTe are deposited on the ZnO surface [23, 24]. The problems of growing such structures are primarily related to the high chemical activity of the synthesised films, which leads to a decrease in the photoactivity of the surface. The solution to this problem can be the application of thin layers of oxides, which perfectly protect the surface from photocorrosion [25].

The synthesis of complex cadmium-tellurium oxides, which exhibit the properties of direct-band semiconductors, was reported [26]. On the other hand, such oxides are often present on the surface in the amorphous phase and exhibit the properties of transparent glass [27]. This makes cadmium-tellurium oxides interesting from the point of view of applying them to the surface of heterostructures. In this aspect, the methods of their synthesis, as well as the possibility of controlling the surface and bulk properties of materials by adjusting the O content in the compounds, require research.

Synthesis methods are crucial to ensure the surface morphology, crystallinity, and composition of nanostructures and thin films [28, 29]. $CdTeO_3$ films were grown using reactive pulsed laser deposition [30]. Cadmium oxides with nanobelt-type morphology were prepared using the sol–gel method, which exhibited excellent photocatalytic properties [31].

Conventionally, the methods for the synthesis of thin films can be divided into physical and chemical methods. Physical methods are characterised by the deposition of film material from the target source onto the surface of the substrate [32]. These methods include vacuum evaporation and sputtering, and high temperature and/or pressure regimes are often used [33, 34]. Chemical methods for synthesis of nanocoatings include chemical deposition from the gas phase, laser CVD, organic chemical deposition of metals (MOCVD), chemical and electrochemical etching, etc. [35-37]. In recent years, the technology of a Successive Ionic Layer Adsorption and Reaction (SILAR) has gained popularity, as it allows the deposit of thin films on large areas. This method can be considered a modification of the chemical precipitation method, with the difference that SILAR is implemented by repeating the reaction steps with intermediate sample washing steps. This technology is inexpensive, as it does not require hightech equipment, a vacuum, and long periods of experimentation [38, 39]. The SILAR method allows for layer-by-layer formation of structural elements of different dimensions with specified properties. The deposition rate and film thickness can easily be controlled by varying the deposition cycles.

In this paper, we report the synthesis of a $Cd_xTe_yO_z$ nanocomposite on the surface of the CdS/ZnO heterostructure by the SILAR method, and we also investigate the morphological, structural, and phase properties of the formed structure.

2 Experiment and materials

CdS samples grown by electrochemical deposition on ZnO monocrystalline plates were used for the experiment. To form the CdS/ZnO structure, the cadmium chloride solution prepared according to the following recipe was used: $0.1 \text{ M CdCl}_2 + 0.1 \text{ M CH}_4\text{N}_2\text{S} + 5 \text{ M NH}_3$. The mixture was dissolved in 100 ml of distilled water and stirred with a magnetic stirrer for 20 min at a temperature of 80 °C. The ZnO sample was immersed in the prepared solution and kept for 5 h. After the experiment, the samples were washed in distilled water. It should be noted that the surface of CdS after electrochemical deposition has a large number of broken bonds, which leads to partial oxidation with the formation of CdO cadmium oxide.

The $Cd_{x}Te_{y}O_{z}$ oxides on the CdS/ZnO substrate were formed by the successive ionic layer adsorption and reaction method (SILAR) during cyclic treatment in precursor solutions with subsequent removal of excess reagent from the substrate surface by washing in H₂O₂ (Fig. 1). The number of processing cycles is 5. The use of five processing cycles ensures that sufficient time and reagents are available to precipitate the complex oxide products that form in the chemical reaction between Te and Cd ions. In addition, the number of processing cycles depends on the amount of material required for a thin uniform film evenly distributed over the substrate. Based on our empirical data and literature sources, it was established that a greater or lesser number of processing cycles will lead to the formation of a film that is too thick or too thin, which may be unevenly distributed over the surface [39]. One cycle consisted of four stages:



Fig. 1 Schematic illustration of the formation of thin films in the SILAR method

- 1 stage. Immersion of the samples in the precursor aqueous solution of 0.01 M Na₂TeO₃, processing time— 10 min. The solution was heated to 50 °C. During this stage, Te ions are adsorbed on the surface and enter a chemical reaction with the substrate ions.
- 2nd stage. Washing of the samples in H₂O₂ hydrogen peroxide to remove excess tellurium atoms and reaction products. Processing time—2 min.
- 3rd stage. Immersion of the samples in the precursor alcohol solution of 0.01 M Cd(NO₃)₂. The temperature of the solution is 50 °C. During this stage, Cd ions are adsorbed on the surface, and $Cd_xTe_yO_z$ compounds are formed.
- 4th stage. Repeated washing of the samples in H₂O₂ to remove weakly bound ions for 2 min.

For better adsorption and adhesion, electrolytes were stirred with a magnetic stirrer at low speed during deposition. After completing five cycles of processing the samples using the SILAR method, annealing was carried out in a JetFirst diffusion furnace for 20 min at a temperature of 150 °C. The annealing was carried out in atmospheric air to stabilise the surface states due to saturation of the surface with O. This regime was chosen because this temperature is effective in stabilising surface states due to saturation of the O surface and will not allow the evaporation of other elements. Furthermore, on the basis of our own empirical studies, it was established that 20 min is the minimum time required to stabilise the surface states of the $Cd_xTe_yO_z$ nanocomposite. After that, the samples were kept outdoors for 3 months.

The morphology of the films was studied using the SEO-SEM Inspect S50-B microscope at 20 kV. Elemental analysis of the surface was performed using energy dispersive spectrometry. Quantitative evaluation of the spectra was carried out in the self-calibrating detector mode. X-ray diffraction (XRD) and RAMAN spectroscopy were used to analyse the phase composition and structural and substructural parameters.

3 Results and discussion

3.1 XRD analysis

XRD spectra were measured in the angle range of 20–80. This range will be chosen as it corresponds to the typical range of diffraction angles for crystalline materials CdO, TeO₂, TeO₃, TeO₄, CdTe₃O₈, CdTeO₃, allowing for the identification of the crystal structure and determination of lattice parameters (Fig. 2). The XRD patterns of the Cd_xTe_yO_z films grown by the SILAR method and theoretical patterns CdO, TeO₂, TeO₃, TeO₄, CdTe₃O₈, CdTeO₃ are shown in Fig. 3. The presence of peaks of binary tellurium oxides (TeO₂, TeO₃, TeO₄) and ternary cadmium-tellurium oxides (CdTe₃O₈, CdTeO₃) can be observed. The most intense peaks correspond to CdTe₃O₈. This indicates the predominance of this oxide over others in the formed nanocomposite.



Fig. 2 Theoretical peaks XRD for CdO (a), TeO₂ (b), TeO₃ (c), TeO₄ (d), CdTe₃O₈ (e), CdTeO₃ (f) (obtained from VESTA)

Fig. 3 XRD spectra of nanocomposite $Cd_xTe_yO_z$



Table 1 Crystal structures determined by XRD

No	2 <i>θ</i> , °	hkl	Oxide	Crystal system
1	50.05	(220)	TeO ₃	Trigonal
2	20.1	(111)	CdTe ₃ O ₈	Monoclinic
3	26.35	(310)		
4	27.85	(112)		
5	33.95	(020)		
6	26.5	(111)	TeO_4	Monoclinic
7	36.6	(112)		
8	40.34	(020)		
9	31.45	(040)	CdTeO ₃	Orthorhombic
10	54.4	(220)		
11	63.85	(004)		
12	75.5	(110)		
13	29.95	(102)	TeO ₂	Orthorhombic
14	62.05	(024)		

In addition, the intense peak at $2\theta = 26.5^{\circ}$ is associated with the presence of binary oxide TeO₄ (Fig. 3, Table 1). It can be seen that the oxides CdTe₃O₈ and TeO₄ have a similar crystal structure (Fig. 4). It is opinion that TeO₄ is the structural component of CdTe₃O₈ [40]. These oxides crystallise on the surface in a monoclinic crystal lattice and have adjacent space group numbers—13 and 14 for CdTe₃O₈ and TeO₄, respectively.

Similarly, the similarity of the crystal lattices and parameters of TeO_2 and CdTeO_3 (Fig. 5) gives reason to assert the common nature of the formation of these oxides [40, 41]. Their crystal lattices belong to orthorhombic space group numbers, group numbers—61 and 62 for CdTeO_3 and TeO_2 , respectively. The rather small values of the peak width at half-height indicate that the crystallites are nanometre-sized. On the other hand, the existing noise and low intensity of the peaks of TeO_2 , TeO_3 , CdTeO_3



Fig. 4 Crystal lattices and structural parameters of $CdTe_3O_8$ (a) and TeO_4 (b)



Fig. 5 Crystal lattices and structural parameters of $CdTeO_3$ (a) and TeO_2 (b)

indicate surface amorphization. That is, these oxides can be on the surface in the form of transparent glass. It should also be noted that most of the peaks compared to the theoretical ones (Figs. 2, 3) show a shift to the left, which may also indicate the formation of oxide layer in the form of nanometre-sized crystallites.

The very weak peak at $2\theta = 50.05^{\circ}$ corresponds to the trigonal phase TeO₃. Therefore, it is possible to assume that this oxide is formed on the surface of the CdS substrate, the sources of oxygen of which are CdO atoms. Additionally, when O is supplied, TeO₄ is formed by the following reaction:

$$2OH^{-} + TeO_3^{2-} \rightarrow TeO_4^{2-} + H_2O + 2^{e-}$$
.

The following is the attachment of cadmium atoms to the formation of $CdTe_3O_8$. An alternative process is also possible, the eruption of oxygen atoms from TeO_3 . In this case, TeO_3 goes to TeO_2 . When the surface is saturated with Cd atoms, TeO_2 becomes a source of formation of the ternary oxide CdTeO₃. That is, TeO3 is a more energetically favourable form for the formation of tellurium oxides on the surface of CdS covered with CdO nanoparticles. This is explained by a greater correspondence of the TeO_3 parameters of the crystal lattices with CdS and CdO in comparison with other tellurium oxides. With further ionic oxidation of the surface of the sample, there is a transition to other tellurium oxides ($TeO_2 \leftarrow TeO_3 \rightarrow TeO_4$), as described above.

Note that the XRD peaks from CdS, CdTe, and CdO have not been detected. There are several important conclusions from this:

 As a result of the deposition of ion layers on the surface of the heterostructure, CdS/ZNO formed a rather dense nanocompository layer Cd_xTe_yO_z;

- Due to the presence of nanometric particles, partial amorphization occurred both on the surface and on the volume of the structure;
- Most likely, CdO on the CdS surface was present in the form of nanoparticles;
- CdTe was not formed on the surface of the CdS/ZnO heterostructure; instead, oxidation processes with the formation of complexes of $Cd_xTe_yO_z$ oxides took place.

A very good match between the crystal lattice parameters of the surface of CdS and CdTe should lead to the formation of CdTe on the surface of CdS by replacing S atoms with Te atoms. However, this did not happen, which once again confirms the presence of a passivating layer of cadmium oxide on the CdS/ZnO surface. Due to its high chemical activity, the SILAR method allows for the processes of dissolution of the passivating layer with the subsequent formation of new oxide compounds. In this way, the crystal lattices of the surface layers are rearranged, and the material acquires new properties.

3.2 Raman spectra analysis

Figure 6 shows the Raman spectrum of the $Cd_xTe_yO_z$ nanocomposite synthesised on the CdS/ZnO surface. Three characteristic regions can be seen. In the low-frequency part of the spectrum, the presence of medium intensity spectra is observed. The most intense peak is at a frequency of 602 cm⁻¹. In the high-frequency part of the spectrum, the presence of low-intensity oscillatory peaks is observed. In this area, the spectrum also shows a sharp rise, which may be due to the heating of the sample during the recording of the spectra.

All combination modes were identified and listed in the Table 2. In general, the main peaks are attributed to tellurium oxides (TeO_2 , TeO_3 , TeO_4). The most intense peak at

Fig. 6 Raman scattering spectra of the $Cd_xTe_yO_z$ nanocomposite formed on the surface of the CdS/ZnO heterostructure by the SILAR method



Table 2Band assignment fromRaman spectra

$\nu_{\rm c}^{\ a}, {\rm cm}^{-1}$	Band	Assignment	References
109 w	$A_{\rm g}$	Te-O-Te	[41, 43, 45]
	B ₁	Phonon vibrations—Te structure	[44]
145 vw	A_1	Phonon vibrations—Te structure;	[45]
	Е	Intra-chain vibrations of Te-Te bonds	[44, 48]
213 m	<i>E</i> (LO)	TeO ₂	[44, 48]
254 m	ТО	β-CdO	[49]
300 s	$(B_1 + E)$	γ-TeO ₂	[44, 47]
348 w	E (2LO)	TeO ₂ asymmetric deformation	[41, 44]
369 w	D	TeO ₃ asymmetric deformation	[41]
602 vs	D	Stretching vibrations TeO ₄	[43]
	B_1	Asymmetric stretching vibrations in Te–O–Te bridges as in γ -TeO ₂	[44]
739 vw	B_2	Stretching vibrations TeO ₃	[44]
		Non-bridging elongation of Te–O (NBO) bonds in TeO ₃ polyhedra;	
	E (LO)	α-TeO ₂	[47]
		CdTeO ₃	[48]
748 w	<i>E</i> (LO)	α-TeO ₂	[42]
		Non-bridging elongation of Te–O (NBO) bonds in TeO ₄ polyhedra	[44]
		γ-TeO ₂	[47]
750–910 vw	A_1, B_1	γ -TeO ₂ , TeO ₄	[43, 45–47]
	E (TO)	$Cd_xTe_yO_z$	[42, 44, 48]

^avs very strong, s strong, m medium, w weak, vw very weak

602 cm⁻¹ is associated with asymmetric stretching vibrations on Te–O–Te bridges, as in γ -TeO₂ (or TeO₄). An intense peak of 300 cm⁻¹ is also attributed to crystalline γ -TeO₂. This is consistent with the results of previous studies [41–49], as well as with the results of XRD analysis.

In general, the structure of tellurium oxide TeO_2 consists of TeO_4 trigonal bipyramids, TeO_{3+1} polyhedra, and TeO3 trigonal pyramids. In the TeO_{3+1} structure, the subscript indicates that one O atom is a greater distance from the Te atom than the other three [44]. When this atom is

significantly removed, the Te–O bond breaks with the formation of TeO₃ oxide. Such a special configuration of tellurium oxides complicates the interpretation of its spectra. It is often impossible to clearly establish the nature of the peak, which can be interpreted as the contribution of various components of the structure (Table 2) [42–48].

The Raman spectrum clearly demonstrates that during the structural phase transition, TeO₃ transforms into TeO₂, which is compatible with orthorhombic CdTeO₃. We can also see that crystalline TeO₄ is compatible with CdTe₃O₈. Thus, when oxygen and tellurium are attached to the surface of the CdS/ZnO heterostructure, we have two phase transitions: hexagonal \rightarrow cubic \rightarrow trigonal. Furthermore, upon saturation with Cd and O atoms, the trigonal tellurium oxide TeO₃ transitions to the monoclinic and orthorhombic configuration of the crystal lattice (Fig. 7). These phase transitions depend on the ratio of the Cd-O-Te components in the synthesised Cd, Te, O, nanocomposite. Extreme cases are the formation of Cd_xO_z and Te_yO_z oxides at x=0 and y=0, respectively (Fig. 7). Excess O content, as well as the formation of Cd–O–Te bonds in CdTeO₂ and CdTe₂O₈ structures leads to polycrystallisation of the surface and partial amorphization due to the presence of nanometre-sized crystallites. This partly explains the low intensity of the peaks in the Raman light scattering spectra in the range of 700–950 cm^{-1} . It is also consistent with the XRD analysis results presented above.

3.3 SEM and EDX analyses

During the ion deposition of Cd and Te using the SILAR method on a CdS/ZnO substrate, it was possible to obtain films with densely packed globular structure and a structural element size of the order of 70–90 nm (Fig. 8). It can be seen from the figure that the structure consists of several structural phases. Phase 1 is characterised by the presence of nanometre-sized a large concentration of spherical globules. Phase 2 has a loose "cloud structure". Phase 3—White areas on the morphology image, which are characterised by the presence of the nanocomposite. It can be seen that these areas create a grid over the entire surface; shallow craters consisting of the 1st and 2nd phases are located between the walls of the grid.

EDX analysis taken at three different points of the surface (Fig. 9, Table 3) shows slightly different concentrations of elements on the surface. So, at point 1 (Fig. 9), which





Fig.8 SEM image of the surface morphology of $Cd_xTe_yO_z$ nanocomposite material synthesised on CdS/ZnO substrate by the SILAR method

corresponds to phase 1 of Fig. 8, you can see the presence of only Cd, Te, and O atoms. O is in a higher concentration, which may indirectly indicate the presence of TeO_4 and $CdTe_3O_8$ oxides in these areas, which is also evidenced by the higher concentration of Te in relation to Cd. Spectrum 2 shows almost stoichiometric content of cadmium and tellurium atoms, as well as a small amount of sulphur. That is, the structure of phase 2 is characterised by a loose porous morphology, through which the CdS substrate "sees through". Interesting results were obtained for spectrum 3. Here, in addition to the main elements (Cd, Te, O), you can see the presence in small concentrations of Zn and S. That is, in these areas (phase 3, Fig. 8) peaks from Zn are very weak



Fig. 7 a the ratio of Cd, O, and Te elements in the nanocomposite $Cd_xTe_yO_z$; **b** phase transitions in the $Cd_xTe_yO_z/CdS/ZnO$ heterostructure





Fig.9 EDX analysis of the surface of the $Cd_xTe_yO_z/CdS/ZnO$ sample: **a** the surface section for which the analysis of the component composition was performed; **b** EDX spectra taken at three points on the surface of the sample

 Table 3
 Component composition of elements on the surface of the synthesised nanocomposite (atomic units)

Spectrum no	Te, %	O, %	S, %	Zn, %	Cd, %
Spectrum 1	29.49	45.73	0	0	24.78
Spectrum 2	28.76	38.37	5.13	0	27.74
Spectrum 3	21.42	35.52	9.39	3.55	30.12

and appear as a result of "transmission" of a thick layer of ZnO through the structure of CdS, CdO, $Cd_xTe_yO_z$.

From these results, it is possible to predict the growth mechanism of the $Cd_xTe_yO_z$ oxide layer on the CdS/ZnO surface (Fig. 10). As a rule, loose, uneven layers with nanocrystallites separated from each other grow on the surface of the substrate according to the Volmer–Weber mechanism. The Volmer–Weber mechanism is a growth mode in which the film grows by forming isolated islands on the substrate [50]. With further growth, the islands can merge until a continuous film forms. This film growth corresponds to a situation in which Te–O and Cd–O–Te bonds are stronger than those





with the substrate. This issue requires further research to improve the synthesis technologies of high-quality $Cd_xTe_yO_z$ oxide passivating films, which already look very promising today because of their chemical stability and transparency.

Thus, for the first time, we demonstrated the production of a complex $Cd_{y}Te_{y}O_{z}$ nanocomposite with a densely packed globular structure of nanometre size by the SILAR method. The feasibility of using a CdS/ZnO substrate was also demonstrated for the first time. However, it should be noted that the Cd_xTe_yO_z nanocomposites obtained on the surface of the CdS/ZnO heterostructure by the SILAR method show heterogeneity in morphology and chemical composition and contain many phases. This indicates that the SILAR method exhibits uncontrollability in our case. However, this method is inexpensive, simple, and allows obtaining structures in large sample areas. Therefore, more research is needed on technology for the synthesis of complex composites using the SILAR method, which will allow the formation of materials with predetermined characteristics. Such studies are necessary, first of all, due to the prospects of using $Cd_rTe_vO_z$ films in the creation of dosimeters, phosphors, sensors, and solar batteries [51–54].

4 Conclusions

In summary, our study reports the successful synthesis of $Cd_xTe_yO_z$ nanocomposite films using the SILAR method on a CdS/ZnO surface. Films were grown by repeating deposition cycles using different compositions of ionic electrolytes. Then, the annealing mode in the oxygen flow was applied to complete the process of oxidation of the surface of the heterostructure. As a result, it was established:

The study of XRD and RAMAN spectra made it possible to establish that binary oxides of tellurium (TeO₂, TeO₃, TeO₄) and ternary cadmium-tellurium oxides (CdTe₃O₈, CdTeO₃) are present on the surface of the structure. It was established that TeO₃ is present in the trigonal phase, CdTe₃O₈ and TeO₄ are in the monoclinic phase, and CdTeO₃ and TeO₂ are in the orthorhombic phase. From this, we can conclude that in the first stages of ion deposition, TeO₃ is formed, which is subsequently transformed into TeO_2 (compatible with orthorhombic CdTeO₃) and TeO₄ (compatible with CdTe₃O₈).

In addition, oxides are present on the surface and in the amorphous phase, which may be caused by the presence of nanometer-sized particles and the imperfection of the synthesis technology. Therefore, further studies are needed to establish optimal conditions for the synthesis of $Cd_xTe_yO_z$.

SEM and EDX analysis also showed the presence of three phases on the surface, namely: (1) densely packed globular structure with the size on structural elements of the order of 70–90 nm; (2) loose "cloud structure"; (3) network system of branched pores on the surface of the nanocomposite. Film growth was assumed to occur according to the Volmer–Weber mechanism, which is characteristic of cases of significant mismatch between the crystal lattices of the substrate and the deposited material. The first priority is the growth of TeO₃ oxide, which, when saturated with oxygen atoms, turns into TeO4 or loses oxygen to form TeO₂. When Cd atoms join TeO₄ and TeO₂, they form the oxides CdTe₃O₈ and CdTe₃O₈, respectively.

Overall, this study provides information on the growth mechanisms and structural properties of $Cd_xTe_yO_z$ nanocomposite films, which may have potential applications in the fabrication of dosimeters, phosphors, sensors, and solar cells.

Author contributions Conceptualization, YS; methodology, YS, SK, IB and ZTK; software, YS and SK; validation YS and AIP; formal analysis; investigation, YS, VP, and AIP; resources, YS, IB, ZTK and AIP; writing—original draft preparation, YS; writing—review and editing, YS, SK, VP and AIP. All authors have read and agreed to the published version of the manuscript.

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Data availability Data which are required to supporting finding of this study are present in the article.

Declarations

Conflict of interest The author declares no conflict of interest regarding the publication of this paper.

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