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## Investigation of degradation processes in perovskite under the influence of external factors

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### ABSTRACT

The article describes a number of new fundamental knowledge about mechanisms of degradation processes occurring in photoactive perovskite materials based on complex lead halides and solar cells based on them, modern methods and approaches to increasing the operational stability of perovskite photovoltaic devices are considered. The revealed paths of degradation processes occurring in complex metal halides (lead and tin) under the influence of light and elevated temperatures are important for further developments in the field of creating highly efficient and stable perovskite solar cells of a new generation. The investigated models of degradation are described both under the action of moisture and as a result of radiation ionization processes. The importance of solving the Dexter-Varley paradox, which takes into account the competition between the processes of displacement of ISO states, as well as the delocalization of the resulting hole in the valence band, is emphasized. It was shown that by changing the force of pressure of the tape on the perovskite film, it was possible to achieve the maximum values of the light conversion efficiency of about 12.7%. It was found that the presence of charge carriers in the form of polarons can significantly affect the assessment of the degradation efficiency towards its increase. The data obtained can radically change the traditional ideas about the efficiency of photochemical reactions.

**Keywords:** perovskite materials, degradation processes, encapsulation.

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## Introduction

The development of hybrid complex lead halides with a perovskite structure is the most promising area of research in the field of creating a new generation of thin-film phototransformers. The modern approach to the processing of mineral raw materials should be based on the use of effective technological methods that ensure the integrated use of raw materials and the environmental friendliness of the processing process [1]. These materials demonstrate impressive optoelectronic properties, and solar cells based on them – record growth rates of light conversion efficiency. A significant disadvantage of hybrid perovskites is their low resistance to various external (oxygen, moisture) and internal (electric field, temperature, light) factors. It should be emphasized that the influence of external factors is the least dangerous

for these materials, which is eliminated by isolating the photoactive layer of the solar battery with special encapsulating materials. The same approach of PSC stabilization by encapsulation is practically not applicable to protect devices from the influence of internal factors. In this regard, the researchers are faced with the task of finding ways to prevent the degradation of perovskite materials caused by internal factors. Various approaches have been proposed for the stabilization of devices based on complex lead halides, from optimizing the chemical composition of perovskite films by introducing modifying additives into the perovskite layer to optimizing the architecture of perovskite solar cells. However, for the targeted development of effective approaches to improving the stability of perovskite systems and solar batteries based on them, a deeper understanding of the processes of photochemical and thermal degradation of various

types of complex metal halides is necessary. At the same time, to obtain reliable data, it is necessary to study the degradation processes of perovskite materials under strictly controlled conditions in an inert atmosphere.

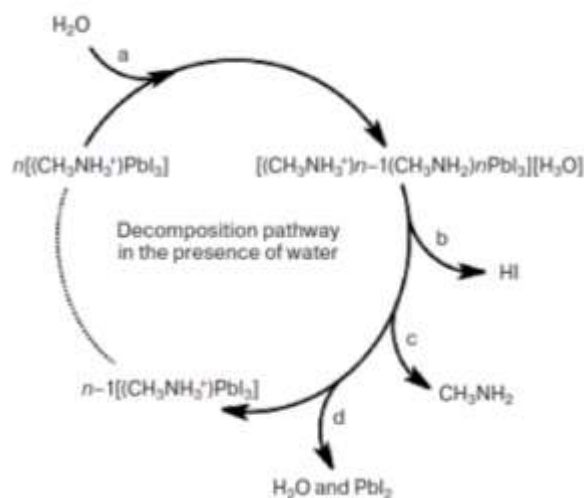
**The problem of stability of perovskite solar cells.** For the production of efficient and at the same time cheap solar panels based on perovskites, it is necessary that the solar panels have long-term operational stability. To date, the warranty period of silicon solar cells is 20-25 years. If the efficiency of a perovskite solar battery is reduced by 1% every year, then in 20 years it will degrade by 20%, which is quite an acceptable indicator in the field of photovoltaics [2]. Just like organic solar panels, devices based on perovskite materials have significant stability problems. This is due both to the true instability of the perovskite material itself, and to external factors that have a detrimental effect on the device as a whole.

**External factors contributing to the degradation of perovskite materials.** Despite the enormous advantages of perovskite solar cells over other photo-converters, these types of devices, unfortunately, are still far from practical implementation. The main problem is the extremely low stability of complex lead halides. Halide (especially iodide) complexes of lead are rapidly destroyed under conditions of elevated temperatures, as well as under the influence of moisture and oxygen in the air [3,4]. In addition, perovskite materials based on lead halides are characterized by low photochemical and electrochemical stability [5,6]. In real-world operating conditions, the efficiency of solar cells based on  $\text{MAPbI}_3$  drops to almost zero in just a few hours, which makes it impractical to use them in practice.

### Experimental part

**Degradation effects in perovskites.** The problem of degradation of solar cells based on organic-inorganic perovskites is very acute in two respects. First, like every salt, the interaction with moisture is very significant here. Secondly, the ionic nature of the lattice and the large value of the Madelung energy open up specific pathways for photochemical reactions. Let us briefly consider these two features. In the infusional hypothetical scheme of the influence of moisture on the decomposition of perovskite has now been elucidated (see Figure 1).

Figure 1 shows that when one water molecule interacts with  $n$  "molecules" of perovskite, one water molecule and one  $\text{PbI}_2$  molecule are separated in several successive stages. This scheme goes back to the Grothaus mechanism [7], so that the molecules  $\text{HI}$  and  $\text{CH}_3\text{NH}_2$  are still isolated along the path of the complete reaction.



**Figure 1** - Possible decomposition pathway of hybrid halide perovskites in the presence of water

One water molecule is required to initiate this process, with the decomposition being driven by the phase changes of both hydrogen iodide (soluble in water) and the methylammonia (both volatile and soluble in water) [8].

Apparently, it is still not clear how to change the composition of perovskite to reduce degradation under the influence of moisture, both in the presence and in the absence of photoexcitation of the material.

It is necessary, however, to keep in mind that there are other (without moisture) channels of degradation of perovskite under the influence of radiation. One such path is indicated in [9], who believes that after the generation of a hole on the iodine ion, this hole migrates to the surface, after which it is on the surface that the reaction of neutral iodine with one of the surface negative iodine ions occurs. This position is the originator of a whole sequence of reactions, resulting in the disintegration of the perovskite structure. However, when analyzing this scheme, an extremely important circumstance is not taken into account: the passage of the reaction between neutral iodine and a negatively charged iodine ion is prevented by the delocalization of the hole, which in the zone scheme is the neutral iodine atom. Taking into

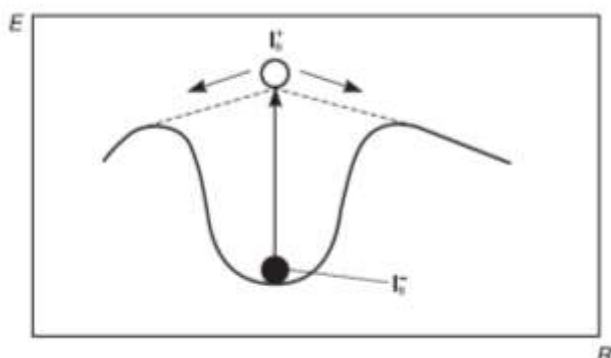
account this circumstance requires a special analysis (namely, the resolution of the Dexter-Varley paradox), which is particularly clearly manifested in the problems of subthreshold defect formation and has been studied since the late 50s of the XX century. In our recent work on the ionization-stimulated degradation of perovskite, this problem was studied in some detail [10]. Indeed (Fig. 2), if a photon tears an electron from any nodal atom of an iodine ion, this means the simultaneous manifestation of two circumstances: first, if we proceed from the band structure, the ionization of iodine means the appearance of a hole in the valence band; second, from the point of view of crystal chemistry, the neutralization of iodine immediately leads to the elimination of a potential Modelung pit for it (the depth of this pit was calculated earlier (see Table 1).

**Table 1** - Calculated properties of four hybrid lead halide perovskites from density functional theory

Cation	D, D	a, nm	$\Delta P, \mu C / cm^2$	$E_{rot}, kJ / mol$	$E_{dip}, kJ/mol$
NH <sub>4</sub>	0	0.621	8	0.3	0
CH <sub>3</sub> NH <sub>3</sub>	2.29	0.629	38	1.3	4.6
CF <sub>3</sub> NH <sub>3</sub>	6.58	0.635	48	21.4	42
NH <sub>2</sub> CH <sub>3</sub>	0.21	0.634	63	13.9	0.03

Note. The electron polarization of the lattice ( $\Delta P$ ) and the rotational barrier energy ( $E_{rot}$ ) were calculated by PBEsol in VASP. The nearest neighbor dipole interaction ( $E_{dip}$ ) was estimated from the point dipole calculation (source: [8]).

After the elimination of the Modelung pit, the neutral iodine atom appears on the hump of the crystal potential and tends to slide into the neighboring internode.



**Figure 2** - The formation of dynamic Coulomb instability in the I-sublattice of perovskite [10]

This takes a time of  $\tau_+ \approx 5 \cdot 10^{-14}$  s. However, the hole is localized at the node also during the finite time  $\tau_e$ . If  $\tau_e$  is much less than  $\tau_+$ , then the Dexter displacement of the iodine atom in the internode does not have time to occur, so there will be no defect formation (degradation). However, the situation is much subtler: the motion of the hole is a quantum process, which means that there are both faster and slower cases of delocalization of the hole (for  $\tau_e$  is the average value). Therefore, to find the probability of displacement of iodine atoms in the internode, despite the competition of hole delocalization, it is necessary to do a quantum calculation. This gives for the probability of displacement of internode ions the probability of the following type:  $n = \exp(-\tau_+ / \tau_e)$ . The estimation of the value of  $\tau_e$  is possible on the basis of the uncertainty ratio:  $\tau_e = h / \Delta E v$ , where  $\Delta E v$  is the width of the valence band formed from the P states of iodine. For the parameters of perovskite (CH<sub>3</sub> – NH<sub>3</sub>)PbI<sub>3</sub>, the characteristic value of the probability of displacement of the iodine atom in the internode is  $n \approx 10^{-4}$ , which means that after ionization of the iodine ion, one of 10,000 ionizations changes this determines the degradation ability of the crystal. Obviously, it is by a factor of this order that the entire Schumann scheme is reduced.

**Encapsulation as an effective method of protecting the photoactive layer of solar cells from external factors.** Encapsulation is a technology for protecting solar cell components from the influence of aggressive environments (O<sub>2</sub>, H<sub>2</sub>O) by using an impermeable capsule. Typically, thin plates of glass and epoxy glue are used for encapsulation, which hardens under the influence of ultraviolet light (Figure 3) [11-13]. Han and colleagues investigated two different methods of encapsulation of perovskite solar cells. [14]. The first method was to use a UV-curable adhesive between the top silver electrode of the device and a regular slide. In the second method, a technique was used in which the insulation of the active layer was carried out using a special glass "lid", in the niche of which a desiccant was located, which acts as an adsorbent of moisture, in the case of its penetration through the epoxy glue [15].

This approach has further improved the efficiency and stability of the devices. Figure 3. Schematic illustration of perovskite solar cell encapsulation. However, such types of encapsulation are unlikely to be applied on an industrial scale. For perovskite solar cells, the protection methods used for organic solar cells



**Figure 3** - Schematic illustration of perovskite solar cell encapsulation

manufactured by roll technologies can be very effective. In 2009, Krebs et al. developed a technology for the production of fully encapsulated flexible polymer solar cells [16].

The substrate in such batteries is polyethyleneterephthalate (PET), and the encapsulation was carried out by laminating a thin film of 25 microns of the same PET with an acrylic adhesive applied. As a result, this technique has led to a significant increase in the stability of devices in real-world operating conditions of solar panels. The disadvantage of the developed method was the degradation of the manufactured devices in the places of the roll cut. But this problem is easily eliminated by treating the edges with a special sealant. Another interesting and inexpensive way to increase the stability of devices by encapsulation was to use a copper tape instead of the usual sputtered top electrodes.

The conductive copper adhesive tape is pasted over the photoactive layer using a conventional glass rod. By varying the pressure force of the tape on the perovskite film, it was possible to achieve the maximum values of the light conversion efficiency of about 12.7%. Devices obtained by this method also showed higher stability in air compared to devices with sputtered electrodes. However, it is worth emphasizing that

encapsulation protects the active layer of a perovskite solar cell only from the effects of aggressive environments, but cannot prevent the influence of internal factors such as electric field, elevated temperature, and sunlight. Therefore, the study of the influence of these factors on the stability of perovskite solar cells requires serious and detailed consideration.

## Conclusions

The article describes in detail the degradation models (both under the influence of moisture and as a result of radiation ionization processes). The role of solving the Dexter-Varley paradox (taking into account the competition between the processes of ISO state displacement and delocalization of the resulting hole in the valence band) is emphasized; it is shown that the latter circumstance can radically change the ideas about the efficiency of photochemical reactions. It should be noted in this connection that the existence of charge carriers in the form of polarons can have a significant impact on the assessment of the efficiency of degradation in the direction of its increase.

The publications of recent years not only reflect the state and problems, but also reasonable hopes that the future of alternative energy sources lies with perovskite-based cells.

**Conflict of interests.** On behalf of all authors, the correspondent author declares that there is no conflict of interests.

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## Сыртқы факторлардың әсерінен болатын перовскиттегі деградациялық процестерді зерттеу

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<p>Мақала келді: 06 мамыр 2021 Сараптамадан өтті: 07 маусым 2021 Қабылданды: 05 қазан 2021</p>	<p><b>ТҮЙІНДЕМЕ</b></p> <p>Мақалада күрделі қорғасын галогенидтері мен оларға негізделген күн батареялары негізінде фотоактивті перовскит материалдарында болатын деградация процестерінің механизмдері туралы бірқатар жаңа іргелі білім келтірілген. Перовскиттік фотоэлектрлік қондырғылардың жұмыс тұрақтылығын арттырудың заманауи әдістері мен тәсілдері қарастырылған. Жарық пен жоғары температураның әсерінен металдардың кешенді галогенидтерінде (қорғасын мен қалайы) жүретін деградация процестерінің анықталған жолдары жаңа бұйындық жоғары тиімді және тұрақты күн батареяларын құру саласында жаңа зерттеулерді одан әрі дамыту үшін маңызды. Ыдыраудың зерттелген модельдері ылғалдың әсері кезінде де, радиациялық иондалу процестерінің нәтижесі арқылы да суреттеледі. ISO күйлерінің орын ауыстыру процестері арасындағы бәсекелестікті, сондай-ақ валенттік диапазонында пайда болған тесіктің делокализациясын ескеретін Декстер-Варли парадоксын шешудің маңыздылығына баса назар аударылады. Перовскит пленкаға таспаның қысым күшін өзгерту арқылы жарық конверсиясының ПӘК -нің максималды мәніне шамамен 12,7 % жетуге болатындығы көрсетілді. Полярон түріндегі заряд тасымалдаушылардың болуы оның жоғарылауына қарай деградация тиімділігін бағалауға айтарлықтай әсер етуі мүмкін екендігі анықталды. Алынған мәліметтер фотохимиялық реакциялардың тиімділігі туралы дәстүрлі идеяларды түбегейлі өзгерте алады.</p> <p><b>Түйін сөздер:</b> перовскит материалдары, деградациялық процестер, инкапсуляция.</p>
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## Исследование деградационных процессов в перовските при воздействии внешних факторов

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<p>Поступила: 06 мая 2021 Рецензирование: 07 июня 2021 Принята в печать: 05 октября 2021</p>	<p><b>АННОТАЦИЯ</b></p> <p>В статье описан ряд новых фундаментальных знаний о механизмах деградационных процессов, протекающих в фотоактивных перовскитных материалах на основе комплексных галогенидов свинца и солнечных батареях. На их основе, рассмотрены современные методы и подходы к повышению эксплуатационной стабильности перовскитных фотовольтаических устройств. Выявленные пути деградационных процессов, протекающих в комплексных галогенидах металлов (свинца и олова) под действием света и повышенных температур, имеют важное значение для дальнейших разработок в области создания высокоэффективных и стабильных перовскитных солнечных батарей нового поколения. Исследованные модели деградации описаны как под действием влаги, так и в результате процессов радиационной ионизации. Подчеркивается значение решения парадокса Декстера-Варли, где учитывается конкуренция между процессами смещения ISO-состояний, а также делокализация образующейся дырки в валентной зоне. Показано, что изменяя силу давления ленты на перовскитную пленку, можно было достичь максимальных значений эффективности преобразования света около 12,7 %. Установлено, что наличие носителей заряда в виде поляронов может существенно повлиять на оценку эффективности деградации в сторону ее увеличения. Полученные данные могут коренным образом изменить традиционные представления об эффективности фотохимических реакций.</p> <p><b>Ключевые слова:</b> перовскитные материалы, деградационные процессы, инкапсуляция.</p>
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