

Dear Editor,

We like to express our appreciation to the reviewers for their comments. We are resubmitting the revised version of the paper number JMSE-D-22-00483. We have studied the comments of the reviewer carefully, and have changed the text according to the comments they have listed. The location of revisions is highlighted by yellow in “Marked-JMSE-D-22-00483.pdf”. Below we refer to each of the reviewer’s comments.

## Response to Reviewer #1

**Comment 1.** *Since Ultrasound Stimulated (US) dissociation of FeB pairs in Silicon has been studied before, the author needs to give a short description of the work of reference 16 in the “Introduction” part.*

**Reply:** In fact, the possibility of the ultrasound to change the state of FeB was shown previously [1, 2]. In particular, the FeB pair was revealed [2] to be dissociated in Cz-Si by the action of ultrasound with acoustic strain  $\xi_{\text{US}} = 10^{-5}$ – $10^{-4}$ . Furthermore, Ostapenko and Bell [2] regarded the resonance condition of pair dissociation and used 25–70 kHz. Besides, it was asserted [1] that in the case of predominant dissociated pairs, the ultrasound may promote the pairing reaction in contradistinction to the case of a high fraction of paired iron. But empirical evidence for this prediction is absent. In this work, i) the  $f_{\text{US}} = 2$ – $30$  MHz and subthreshold strain  $\xi_{\text{US}} < 2 \times 10^{-6}$  were used, which were deficient to effectively overcome the Coulombic attraction between  $\text{Fe}_i^+$  and  $\text{B}_s^-$ ; ii) the predominant dissociation of FeB was realized by intense illumination. Thus the association of FeB pair (the migration of  $\text{Fe}_i^+$ ) was firstly investigated experimentally in conditions of USL.

The additional information was added to the revised manuscript (last paragraph in “Introduction”).

**Comment 2.** *The font in Figure 4 is obviously too small.*

**Reply:** The font was enlarged.

**Comment 3.** *The forms of the separation of axis titles and units are inconsistent in the Figures.*

**Reply:** All axis titles were changed according to “Axis title (unit)”.

## Response to Reviewer #2

**Comment 1.** *Abstract: This part is precise and concise. It could be better to mention some points of the light induced degradation in solar cells.*

**Reply:** The different light-induced degradation (LID) phenomena exist that affect the efficiency of Cz-silicon solar cells due to a decrease in lifetime of generated excess charge carriers. The main reasons for this transformation are boron–oxygen complex formation (BO-LID) [3] and iron–boron pair

dissociation (FeB-LID). Besides, the light- and elevated-temperature-induced degradation (LeTID) is observed. In recent studies the occurrence of the LeTID defect could be related to the presence of hydrogen and metal impurities [4–6]. We study the influence of ultrasound on LID recovery. The complete recovery in the dark at near room temperature and the determined value of activation energy (0.656 eV) evidenced the iron–boron pair LID in our case.

The text was revised (Abstract and page 4, paragraph 2).

**Comment 2.** *Introduction: Why not other doping elements ? why iron given more importance ? explanation required. Objective is not well framed.*

**Reply:** The silicon solar cells (SCs) constitute about 90% of current global photovoltaic production capacity. Iron is one of the most relevant, omnipresent, and efficiency-limiting metallic impurities in p-type silicon solar cells [7, 8]. Therefore, the methods of defect engineering aimed at iron have practical importance. On the one hand, shallow acceptors (B, Al, Ga, In) are effective trapping sites for iron around room temperature and in darkness in p-Si due to electrostatic attraction between the negatively charged acceptors and the positively charged iron ions. All Fe-acceptor pairs are similar: complexes have two structural configurations with trigonal and orthorhombic symmetry and can be broken by intense illumination and/or annealing above 200°C [7, 9]. On the other hand, the back surface field (BSF) and passivated emitter and rear cell (PERC) are the most popular designs that have been used in the mass-production of Si SCs, and both BSF and PERC are mainly based on boron-doped silicon wafers [10, 11]. Therefore the iron–boron pair is one of the most relevant complexes to the defect engineering in real SCs. The aim of this work is to investigate experimentally how acoustic waves (AWs) influence the ability of iron to diffuse in silicon SCs.

The text was revised (second and third paragraphs in “Introduction”).

**Comment 3.** *Experimental part: How much doping of phosphorus? How do they measure thickness?*

**Reply:** A diffusion from the gas phase ( $\text{POCl}_3$ ) at 940°C was performed on wafers resulting in a  $n^+$ -emitter layer on the front side (sheet resistance of about 20 – 30  $\Omega/\square$ , thickness of 0.7  $\mu\text{m}$ ). In addition, to reduce recombination losses and increase the conductivity of the contact layer, a  $p^+$  layer (10 – 20  $\Omega/\square$ , 0.6  $\mu\text{m}$ ) was formed by boron diffusion from the gas phase ( $\text{BCl}_3$ ) at 985°C on the rear surface. The solid and grid aluminum contacts were formed by magnetron sputtering on the rear and front surfaces, respectively.

The layer thicknesses were evaluated by using an annealing temperature and duration as well as an partial pressure gas.

More detailed information about samples was added (first paragraph in “Experimental and Calculation Details”).

**Comment 4.** *Why were AWs excited in the samples of 2.4; 4.1; 5.4; 9.0; 14; 18; 31 MHz (longitudinal) or 0.3 MHz (transverse) with frequency  $f_{US}$ ?. Give a reason. Why not selected substitution atoms?*

**Reply:** We have previously shown [12–17] that waves with megahertz frequency can be an effective tool for defect engineering in silicon structures. Thus in this work, we tried to use such waves to control iron impurities in silicon SCs.

On the other hand, it is widely known that the efficiency of ultrasound influence on defects in semiconductors depends on acoustic wave frequency. Moreover, the type of frequency dependence is determined by the mechanism of acousto-defect interaction [18–20]. The set of longitudinal wave frequencies (2.4; 4.1; 5.4; 9.0; 14; 18; 31 MHz) was used to establish features of ultrasound influence on iron migration. Besides, the effect of the increase in the carrier capture coefficient for defects in silicon SC is shown [12] to be intensified in the case of the transverse acoustic waves using. Accordingly, the transverse waves (0.3 MHz) were used as well. In the case of acoustically-induced (AI) change of capture coefficient, the USL causes the variation in the complex components' distance [12]. In our case, the AI effect is the opposite and is weakened for transverse waves using. Therefore, the acceleration of the FeB pair association does not deal with iron-boron distance change.

The text was revised (second paragraph in “Experimental and Calculation Details”; page 9, paragraph 1).

**Comment 5.** *Result and discussion: In figure 5,  $x$  axis when will it reach minimum? Is there any particular reason for selecting 300 to 304K?*

**Reply:** The investigations have shown that the dependence of  $\tau_{US}/\tau_0$  on ultrasound intensity is close to linear at low  $W_{US}$ . The increase in  $W_{US}$  leads to saturation, which correspond to  $\tau_{US} \simeq 0.7\tau_0$  at  $T = 340$  K. Therefore the minimum (saturation) of curves in Fig. 5 is expected at about  $0.3 \text{ W/cm}^2$ . It should be noted that according to Ostapenko and Bell [2], the further increase in acoustic strain up to  $\xi_{US} = 10^{-5}$  may lead to FeB pair dissociation and increase in  $\tau_{US}$ .

The temperature range is limited for the following reasons. On the one hand, the investigations have shown that the ultrasound influence on FeB pair association time weakened with temperature decrease. In fact, the effect value is about 5% only for  $T = 300$  K — see Fig. 6. Therefore the experiments seem useless at  $T < 300$ . On the other hand, the portion of interstitial iron atoms that remain unpaired in the equilibrium state increases with temperature rise. According to Wijaranakula [21], the concentration of unpaired iron atom  $N_{Fei,eq}$  depends on temperature, doping level, and concentration of all iron impurity atoms  $N_{Fe,0}$ . The estimations show that at 340 K  $N_{Fei,eq} \simeq 0.1N_{Fe,0}$ . The further temperature decrease leads to an increase in  $N_{Fei,eq}/N_{Fe,0}$  value and to a significant decrease in change of short circuit current after intense illumination and measurement accuracy of association time.

## Response to Reviewer #3

**Comment 1.** *The results are new and may be considered for publication, however, the presentation of the contents is very poor and hence it is unacceptable in its present form. I guess, the main problem is with the English language used; many unconventional or unusual technical words, which make it difficult to understand what some descriptions mean.*

**Reply:** The text has been revised. We hope for the language improvement.

**Comment 2.** *1. Page 3, line 28, “Fig. 1 Scheme of the sample...” should be changed to, “Fig. 1 Schematic structure of the sample...”. And also in lines 33 -34, this should be changed as, “The schematic structure of SC...”.*

**Reply:** The Reviewer is absolutely right. We have revised the text accordingly.

**Comment 3.** *Fig. 2, presents the measured  $I_{sc}$  as a function of time of illumination  $t$ . Thus the y-axis of Fig. 2 should be labelled as  $I_{sc}$  ( $\mu A$ ) and the x-axis as  $t$  ( $10^3$  s). Also it is not explained what the red and green curves represent? The should explain Red with US and Green without US? Thus, the figure captions of Fig. 2 in lines 26-27 on page 4, should be changed to, “Measured  $I_{sc}$  plotted as a function of the illumination time  $t$ ; red curve- with US and green curve without US. ....”*

**Reply:** The figure captions was revised.

**Comment 4.** *3. The second paragraph, on page 4, lines 36-40, does not make any sense to me. It needs to be rewritten. It should emphasize how  $I_{sc}$  in Eq. (1) is obtained to represent the measure  $I_{sc}$  in Fig. 2.*

**Reply:** The FeB pair association in the dark was accompanied by the  $\tau$  increase and was monitored by measuring the  $I_{SC}$  under LED illumination. The LED illumination induced excess carrier density  $< 10^{12} \text{ cm}^{-3}$ , had duty cycle 0.5% while  $I_{SC}(t)$  measuring, and did not cause FeB dissociation. Moreover, the fitting of the measured dependencies  $I_{SC}(t)$  after high-intensive illumination allows determining the pair concentration and the characteristic time of the FeB complex formation.

The text was revised. (page 5, paragraph 2).

**Comment 5.** *4. In Eq. (1) , please define all the symbols used. For example, what is  $P_{ph}$  ? Also if Eq. (1) represents the measured  $I_{sc}$ , why does it not depend on the time  $t$ ? This needs to be carefully explained.*

**Reply:** The Eq. (1) was modified, all symbols were defined. The parameters, which depend on time, were pointed in Eqs.(2),(3),(6) as well. In initial manuscript the  $P_{ph}$  (LED light power) was introduced on page 3, line 57.

**Comment 6.** Page 5, line 53, "... where  $\tau_{ass}$  is the characteristic time of the complex association." This does not make sense to me. Do you mean, "... where  $\tau_{ass}$  is the characteristic time of the formation of Fe-B complex ?" This needs to be fixed.

**Reply:** We have revised the text accordingly.

**Comment 7.** On page 6, lines 33-37, "... the values of  $\tau_{ass} \dots 1380 \pm 20$  for  $T = 330 \text{ K} \dots 1.26 \pm 0.02)10^4$  for  $T = 300 \text{ K} \dots$ ". Something is wrong here? How can a 30 degree difference in temperature can give such a big value of  $\tau_{ass}$  from Eq. (10).

**Reply:** According to [22–24]

$$\tau_{ass} = 5.7 \times 10^5 \frac{\text{s}}{\text{K cm}^3} \times \frac{T}{N_A} \exp\left(\frac{E_m}{kT}\right), \quad (1)$$

where  $E_m$  is the energy of  $\text{Fe}_i^+$  migration,  $E_m = 0.66 \text{ eV}$  [22–25]. Hence

$$\frac{\tau_{ass}(T = 300\text{K})}{\tau_{ass}(T = 330\text{K})} = \frac{300}{330} \times \exp\left[\frac{0.66}{8.625 \times 10^{-5}} \left(\frac{1}{300} - \frac{1}{330}\right)\right] \simeq 9.2. \quad (2)$$

At the same time

$$\frac{1.26 \times 10^4}{1380} \simeq 9.1. \quad (3)$$

$N_A = 1.4 \times 10^{15} \text{ cm}^{-3}$  for the samples under investigation. Therefore the experimentally determined times are close to ones expected from Eq. (1).

**Comment 8.** Page, 6 lines 53-57, the first sentence of Results and Discussion does not make any sense. Please rewrite it.

**Reply:** The sentence was rewrite in form "The experiments have shown that the US loading leads to speed up of recovery of short circuit current after high-intensive illumination. Therefore, the FeB association is intensified under AW action."

**Comment 9.** Page 6, lines 60-61, "The figure also shows  $\tau_{ass}$  values..." but figure 3 does not show any  $\tau_{ass}$ ? Please check it and fix it carefully.

**Reply:** The text was revised.

**Comment 1.** *The English expression is a little awkward in places, but not to the extent that it negatively affects the readability, in my opinion.*

**Reply:** The text was revised.

## Response to Reviewer #2

**Comment 1.** *In Fig. 4, the numbers identifying the different curves are missing.*

**Reply:** The curves' numbers were added. We apologize for the inattention.

**Comment 2.** *Figure 6 is not so relevant, and could be given as supplementary information*

**Reply:** Figure 6 was transferred to the Supplementary material, the text was revised (page 5, column 1, paragraph 2).

## Response to Reviewer #3

**Comment 1.** *Page 1, column 2, line 1: The lattice deformation amplitude should have a unit.*

**Reply:** The “relative deformation” was meant. The term “strain” is used in the revised version.

**Comment 2.** *Page 1, column 2, paragraph 3: The origin of the iron in the diodes should be explained. How are the diodes contaminated with iron?*

**Reply:** The technological process of solar cells (SCs) manufacturing from Cz-p-Si wafers included the formation of separating and isotype barriers ( $n^+$ - $p$  and  $p$ - $p^+$  junctions) by diffusion of phosphorus ( $\text{POCl}_3$ ) and boron ( $\text{BCl}_3$ ) from the gas phase, respectively; thermal oxidation; thermal annealing; photolithography; etching the dividing groove; chemical treatments; magnetron sputtering of aluminum contacts to the front and back sides.

It has been found that some SC lots have significantly worse parameters compared to typical solar cells for this technological process. In particular, the photoconversion efficiency was almost halved. The analysis showed that the reason for such a deterioration in the SC parameters is a sharp drop in the diffusion length of minority charge carriers (electrons)  $L_n$  in the SC base. Additional experiments with thermal annealing at temperatures of 200°C and 90°C (the procedure is described by Tayyib *et al.*[26]) showed that the decrease in  $L_n$  value is caused by iron impurities available in the SC base at concentrations up to  $4 \cdot 10^{13} \text{ cm}^{-3}$ . It has also been found that the source of iron impurity is insufficiently pure chemicals that were used for chemical treatments in the technological process, obtained from another supplier. These reagents were the source of contamination in the process of manufacturing experimental SC samples.

To study the effect of ultrasonic loading on the kinetics of the transformation of iron-boron pairs, samples with varying degrees of iron contamination (iron concentration  $2 \cdot 10^{12}$ - $4 \cdot 10^{13} \text{ cm}^{-3}$ ) were taken.

More detailed information about iron sources was added (page 1, column 2, paragraphs 3 and 4).

**Comment 3.** *Page 1, column 2, paragraph 5: How was the excess carrier density, which is induced by the LED illumination, estimated? This point should be explained.*

**Reply:** The excess carrier density was estimated by using open-circuit voltage value  $V_{OC}$ . According to Sachenko *et. al.*[27]

$$\Delta n = -\frac{n_0}{2} + \sqrt{\frac{n_0^2}{4} + n_i \exp\left(\frac{qV_{OC}}{kT}\right)} \quad (4)$$

where  $n_0$  is the equilibrium electron concentration,  $n_i$  is the intrinsic electron concentration.

The short information was added to the revised manuscript (page 2, column 1, paragraph 3).

**Comment 4.** *Figure 2: Short circuit current has the unit  $\mu A$  not  $\mu m$ .*

**Reply:** The reviewer is quite right. The graph was corrected.

**Comment 5.** *Page 2, column 2, paragraph 1: The materials doping level is given on page 1 with  $10 \text{ Ohm cm}$ . This is about  $1.4e15 \text{ cm}^{-3}$  and not  $1.4e16 \text{ cm}^{-3}$  as stated here.*

**Reply:** The reviewer is quite right. It was a slip. The correction is done.

**Comment 6.** *Equation 7: The unit of the pre-factor is missing.*

**Reply:** The Equation was corrected.

**Comment 7.** *Page 3, column 1, paragraph 3: The obtained iron concentration is compared to results obtained from diffusion length measurements. There should be a reference to these measurements or more details should be given.*

**Reply:** The diffusion length before ( $L_{n0}$ ) and after ( $L_{n1}$ ) pair dissociation was measured using spectral dependencies of short circuit current Then the iron

concentration was determined by using Zoth and Bergholz[28] equation:

$$N_{\text{Fe}}(\text{cm}^{-3}) = 1.06 \cdot 10^{16} \left( \frac{1}{L_{n1}^2} - \frac{1}{L_{n0}^2} \right) (\mu\text{m}^{-2}) \quad (5)$$

The short information was added to the revised manuscript.

**Comment 8.** *Figure 4: The numbers of the curves given in the caption are not included in the graphs. This must be improved otherwise the figures cannot be understood. Each graph should also be marked by a letter. What is “G” at the x-axis of the inset? This should be explained.*

**Reply:** The graphs were revised. The curves’ numbers were added. Each graph was marked by a letter. “G” was changed by “Will” (radiation intensity of halogen lamp). We apologize for the inattention.

**Comment 9.** *Figure 5: This plot is confusing and must be revised. What is the main statement of this figure? What are the differences between the samples and why do the results change from sample to sample? The axes should have the same scaling. What are the light blue bars in (a)?*

**Reply:** Figure 5 was changed by Table I. The main assignment of this data is an illustration of

- i) USL actually does not influence the  $\tau_{\text{dis}}$  magnitude;
- ii) some pairs do not dissociate under illumination in the USL case when light-induced pair dissociation is close to saturation. The decrease in illumination intensity reduces the last effect at the given illumination times.

The main differences between the samples are the iron concentrations. However, data show that the effect of ultrasound does not qualitatively change with the iron concentration alteration (from sample to sample). The value of acousto-induced change in  $N_{\text{Fe,fit}}$  value depends on ultrasound intensity (see data for sample SC350-1,  $W_{\text{ill}} = 0.16 \text{ W/cm}^2$ ) and frequency.

The dissociation rate of FeB pairs depends on iron concentration, light intensity, and temperature[22, 29–31]. There is a reason of  $\tau_{\text{dis}}$  value change from panel to panel in Fig. 5 in the unrevised manuscript (from row to row in Table I in the revised manuscript)

The light blue bars in Fig. 5(a) (the unrevised manuscript) corresponded to another ultrasound intensity ( $0.6 \text{ W/cm}^2$ ).

**Comment 10.** *Main Problem The impact of ultrasound on the iron-boron-pair reaction was first reported by Ostapenko and Bell in 1995 [1]. They found that the iron boron pairs dissociate due to an ultrasound treatment. This is in contradiction to the findings, which are reported herein. In the contribution under review it is found that the association of the FeB pairs is enhanced by*



the ultrasound treatment. This contradiction must absolutely be discussed by the authors otherwise the manuscript cannot be published.

[1] S. S. Ostapenko and R. E. Bell, "Ultrasound stimulated dissociation of Fe-B pairs in silicon," *J. Appl. Phys.*, vol. 77, no. 10, p. 5458, 1995.

**Reply:** We do not keep silent about reports Ostapenko *et al.* — see Refs.26,27; page 3, column 2, paragraph 1; page 7, column 2, paragraph 1 in the unrevised manuscript. But Reviewer is right and more attention should be paid to the comparison of the findings.

In our opinion, the reported herein and previous results have a lot in common. According to Ostapenko *et al.*[32]  $\text{Fe}_i$  has to "jump" to the next nearest interstitial under ultrasound action; we state about the decrease in  $\text{Fe}_i$  migration energy value as well as enhance of  $\text{Fe}_i$  diffusivity in case of ultrasound loading. The effectiveness of ultrasound influence increase with the rise of temperature in both cases: according to Ostapenko *et al.*, [1, 32] the diffusion length increases from 10.5  $\mu\text{m}$  to 13  $\mu\text{m}$  at 20°C and up to 22  $\mu\text{m}$  at 70°C;  $\Delta E_{\text{US}}$  increases from 3 meV to 8 meV with temperature change from 300 K to 340 K at  $f_{\text{US}} = 0.3$  MHz and  $W_{\text{US}} = 0.76$  W/cm<sup>2</sup> (Fig.6 in the revised manuscript). The difference in the action of ultrasound (the iron-boron pairs dissociation or the enhancing of pairing) is associated with the difference in the intensity of the acoustic influence. For instance, the researchers used acoustic strain  $\xi_{\text{US}} = 10^{-5}$ - $10^{-4}$  was used[2] to dissociate FeB pairs in Cz-Si. Furthermore, Ostapenko and Bell[2] regarded the resonance condition of pair reorientation (first step of dissociation) and used 25 – 70 kHz. In our case,  $\xi_{\text{US}} < 2 \cdot 10^{-6}$  and  $f_{\text{US}} = 2 - 30$  MHz are deficient to effectively overcome the Coulombic attraction between  $\text{Fe}_i^+$  and  $\text{B}_s^-$ . Additionally, the presented data show that the effectiveness of acoustically-induced change decreases as the ultrasound frequency increases.

It should be noted that Ostapenko *et al.*[1] asserted "in the case of predominant dissociated pairs, it [ultrasound treatment] may promote the pairing reaction" (page 1557, column 1, paragraph 2). In our case, the predominant dissociation was realized by intense illumination, and then the ultrasound loading accelerated pairing. Thus, the reported results provided empirical evidence for the above-mentioned prediction.

On the other hand, a certain manifestation of partial acoustically-induced FeB dissociation could be a decrease in the concentration of pairs, which dissociate under illumination in the USL case. In fact, if some pairs were dissociated by ultrasound waves, which had been pre-existed in the sample with a high initial fraction of paired Fe, they can not be dissociated under illumination. The decrease in the temperature or photon quantity leads to reduce in the set of FeB, which was modified by ultrasound, or in the set of FeB, which was modified by the capture of light-induced electrons, respectively. Sets cease to overlap and  $N_{\text{Fe,fit}}(W_{\text{US}} > 0) \simeq N_{\text{Fe,fit}}(W_{\text{US}} = 0)$  — see Table I in the revised manuscript.

Finally, let's summarize the differences between the manuscript and previously reported results. Ostapenko *et al.*[1, 2, 32] investigated the change in

silicon properties (diffusion length) **after** the action of ultrasound with high (above threshold) intensity. This is the so-called ultrasound treatment mode of operation and results can be used to develop a new approach to semiconductor properties modification. Our manuscript is devoted to the modification of the process (short circuit current kinetics or FeB transformation), **during** sub-threshold acoustic wave propagation. This is ultrasound loading mode, which can be used for acoustically controlled tuning of the technological processes.

The additional information was added to the revised manuscript (page 7, column 1, last paragraph; page 7, column 2, paragraph 1).

## References

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