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# Photo- and thermally assisted emission of electrons from rare gas solids

E.V. Savchenko [a,*], O.N. Grigorashchenko [a], A.N. Ogurtsov [a], V.V. Rudenkov [a], G.B. Gumenchuk [b], M. Lorenz [c], M. Frankowski [c,1], A.M. Smith-Gicklhorn [c], V.E. Bondybey [c]

[a] *Institute for Low Temperature Physics and Engineering NASU, Lenin Avenue 47, Kharkov 61103, Ukraine*
[b] *Institute of Radioastronomy NASU, Chervonopraporna str. 4, Kharkov 61002, Ukraine*
[c] *Institut für Physikalische und Theoretische Chemie, TU München, Lichtenbergstraße 4, Garching 85747, Germany*

**Abstract**

Branching of energy relaxation paths in metastable preirradiated solid Ar doped with $O_2$ was studied using a combination of activation spectroscopy methods—thermally stimulated luminescence, thermally and photon-stimulated exoelectron emission. Analysis of the data obtained made it possible to discriminate between surface and bulk electron traps, to elucidate elementary processes involved in the relaxation cascade and to verify the radiation mechanism of energy conversion and transfer from atomic subsystem to electronic one. It was found that recombination of neutral O atoms results in promotion of electrons into the conduction band. A new effect was observed—low temperature desorption from preirradiated solid Ar. It was suggested the effect is due to thermally assisted charge recombination. 

*Keywords:* Noble gases; Electron emission; Desorption induced by electron stimulation

## 1. Introduction

When solid materials, and in particular insulators, are exposed to high energy ionizing radiation, a variety of persistent defects including vacancies, interstitial atoms or ionic centers can be formed. These affect properties of the materials or of their surfaces, and are therefore of a considerable technological importance, and also of interest from the point of view of fundamental surface or solid-state science. The effects of the radiation damage can be reversed for instance by annealing the samples, or by subjecting them to lower energy irradiation. This may stimulate diffusion, recombination of the atoms or charged centers, and in general, relaxation of the high energy defects. Such healing of the defects is often accompanied by spectral changes, light emission or electron emission. Rare gas solids (RGS), either pure, or doped with minute concentrations of suitable guest atoms or molecules, are very simple and theoretically tractable wide gap insulators. They provide very convenient model systems for studying defect formation, and

* Corresponding author. Tel.: +7-380-57-230-0336; fax: +7-380-572-322370/335593.
*E-mail addresses:* savchenko@ilt.kharkov.ua, savchenko@ch.tum.de (E.V. Savchenko).
[1] Permanent address: Institute of Fluid-Flow Machinery, Pol. Acad. Sci., Fiszera 14, 80-231, Gdańsk, Poland.

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the reactions and energy conversion processes accompanying their relaxation. Luminescence which may result from defect relaxation, whether thermally or photon stimulated, is a sensitive tool for its monitoring, as demonstrated by numerous studies of thermally stimulated luminescence (TSL) [1]. This technique was also employed for a study of nominally pure [2–4] and doped [5–10] RGS. Defect relaxation can also cause promotion of electrons into the conduction band, or electron emission from the sample surface. The former process forms the basis for thermally stimulated conductivity (TSC) experiments as recently demonstrated for Ar solids doped with Au, Ag and $O_2$ [8]. The latter process, thermally stimulated exoelectron emission (TSEE) was recently used in our group to investigate charged centers in nominally pure solid Ar [11]. Recent studies of preirradiated RGS revealed that thermally assisted chemical reactions may be involved in the relaxation cascade processes [12].

In this study we present results of an investigation of preirradiated solid Ar samples doped with $O_2$. The defects and their relaxation are monitored using a combination of the TSL, TSEE, and PSEE techniques. As the sample temperature is increased, we observed in addition to electrons emitted by the sample in a weak external electric field, also a desorption of surface atoms which is induced by the charge recombination process. The concurrent use of several techniques provides a better insight into the defect properties and relaxation processes.

## 2. Experimental section

The samples were grown from the gas phase by pulsed deposition on a silver-coated metal substrate cooled by a closed-cycle two-stage Leybold RGD 580 refrigerator. High-purity Ar (99.999%) gas passed through the gas inlet tube directed onto the substrate. Experiments were performed with pure Ar and Ar doped with $O_2$. The dopant to the Ar gas ratio was varied between $10^{-5}$ and $10^{-3}$. A mixture was prepared in a gas-handling system by a standard procedure. Before each experiment, the gas-handling system was degassed by heating under pumping. The pressure in the deposition chamber was $6 \times 10^{-8}$ mbar. The presence of impurities and dopant was monitored by a mass-spectrometer. In order to vary the distribution of the charge carriers and reactive neutral atoms trapped in the samples we used three different procedures of sample preparation: (1) deposition under electron beam (during irradiation of growing film), (2) deposition of neutral gas followed by electron beam irradiation and (3) deposition from discharge. Deposition conditions were varied by changing condensation temperature, gas flow parameters (flow speed, pulse duration and frequency) and electron energy in cases (1) and (2). A hot tungsten filament served as a source of electrons. In case (1) the electrons were accelerated to the energy of 120 eV. The current density of the electron beam was kept at 30 $\mu A\,cm^{-2}$. It enables us to generate charged centers across the whole sample. In case (2) we used an electron beam of energy up to 500 eV to generate carriers in subsurface layer. A typical sample thickness was 50–100 μm. The sample thickness and the deposition rate were determined by measuring the pressure decrease in a known volume of the bulb in the gas inlet system. A typical deposition rate was kept at about $10^{-2}$ $\mu m\,s^{-1}$. Samples of high optical quality were prepared by condensation on a gradually cooled substrate. The first portions of gas were deposited at $T = 70$ K forming layers of more perfect structure. The next layers reproduced the structure to some extent. The procedure of sample preparation from the discharge is described in detail elsewhere [13]. In this case the sample were grown by deposition of a gas from discharge-pulsed supersonic jet on a cooled substrate. Deposition conditions were kept as described above. The advantage of this technique is a high concentration of ionic species and radicals produced by reactions of the metastable rare gas atoms with molecules of dopant in the discharge.

The temperature during irradiation and deposition from the discharge was maintained below the characteristic temperature of the first peak of TSL in solid Ar at $T = 12$ K [3] in order to prevent recombination of charge carriers. The temperature was measured with a calibrated silicon diode sensor, mounted at the substrate. The programmable

temperature controller permitted to keep the desired temperature during deposition and irradiation, as well as to control the heating rate. In the TSEE and TSL experiments samples were heated with a constant rate of 3.2 K $min^{-1}$. The measurements were performed in the temperature range of 7–45 K. The pressure in chamber was measured as a function of temperature simultaneously with current detection.

The emission of electrons from preirradiated samples was detected with a movable Au-coated Faraday plate kept at a small positive potential +9 V. During measurements it was positioned at a distance of 5 mm in front of the sample grown on a grounded substrate. The current from the Faraday plate was amplified by a FEMTO DLPCA 100 current amplifier. The signal was reversed in the polarity by an invertor and digitized in a PC. A current as low as 100 fA can be easily detected.

The total yield of TSL was detected with a PMT. By introducing a convertor of vacuum ultraviolet radiation into visible light we were able to extend the available wavelength range and detect TSL from 800 up to 10 nm.

We also investigated photon-stimulated exoelectron emission (PSEE). In these experiments the samples were held at a temperature (7.5 K) such low that no thermally activated release of electrons from the traps occurred. In solid Ar the threshold temperature of TSEE [11] and TSL [3] is 10 K. In the PSEE experiments a coherent 899-01 dye laser operating with Stilbene 3 and pumped with a coherent Ar ion laser (Innova 200) was used. The laser light was introduced into the sample chamber with an optical fiber. The power did not exceed 20 mW. The emission of electrons was measured with another electrode connected to the same system of current detection.

## 3. Results and discussion

The electronic processes of the relaxation in solids containing localized charge carriers are defined by positions of their local levels with respect to the valence and the conduction bands as well as by sign and mobility of the carriers. The appearance of thermally and photon-stimulated currents is caused by free charge carriers. Mobilities of electrons in all RGS exceed those of holes by factor $10^4$–$10^5$ [14] and direct measurements of charge carrier exit from the surface in the experiments on TSEE [11] showed that the electrons are mobile charge carriers undergone detrapping in solid Ar. Because of negative electron affinity of solid Ar ($E_a = -0.4$ eV [14]) electrons occupying the bottom of the conduction band are at higher energy than the vacuum level, and can escape the surface with corresponding finite kinetic energy. A large electron escape depth of about 500 nm [15] provides experimental access to study of both bulk and surface relaxation processes. Carriers of opposite sign—holes are found to be self-trapped in the lattice in the configuration of rare-gas dimer ions $Rg_2^+$. It was inferred from the theoretical study of hole dynamics [14] as well as from the experiments on laser-induced fluorescence in near infrared range [16–18], measurements of spectrally resolved TSL [2,4,13,19] and photon-stimulated luminescence (PSL) [20] in vacuum ultraviolet range. The most recent results on cluster ions [21] demonstrate also the evolution of charge distribution to a dimer core localization as an increase of cluster size. Based on TSL experiments analysis of photon yield in nominally pure and doped Ar was done previously and parameters of the electron traps estimated [3,4,8,9]. However, the origin of the traps and for some of them even the nature of the parameters are still under discussion [8,11].

The TSL from a standard film of pure Ar gas (Fig. 1a) shows intense maxima at 11 and 15 K. Both of them are characteristic for the matrix and suppressed by doping the sample. The first low temperature peak can be attributed to the release of electrons from the surface traps. The second feature at 15 K is related to radiation induced defects [3,8,9]. Some arguments supporting the assignment will be done below.

In the TSL yield from a standard film of pure Ar gas there are several weak features at about 22 and 35 K. We will focus on the peak at 22 K. There are clear evidences that the oxygen is also involved in the relaxation processes. Doping with $O_2$ enhanced essentially the yield of photons at 22 K in comparison with other peaks and this feature clearly dominates in glow curves of $O_2$ doped

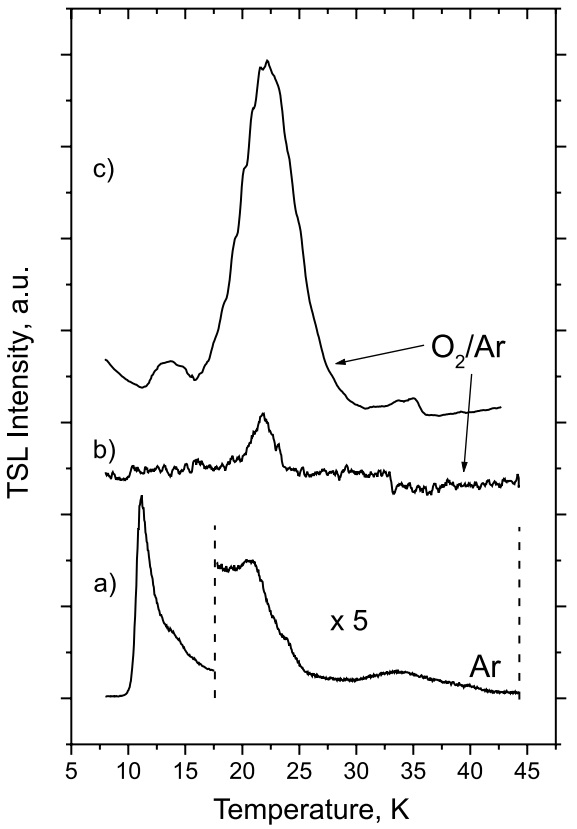


Fig. 1. Total yield of TSL measured from a standard film of pure Ar gas (a) and $O_2$ doped Ar (b, c). The glow curve (c) was measured from the sample grown from discharge, (b) from the sample irradiated by the electron beam. The concentration of $O_2$ in the samples (b) and (c) was about ($10^{-4}$).

Ar (Fig. 1b, c). Interestingly that in glow curve measured from the sample grown from discharge its intensity is extremely high and the half-width of this peak exceeds that of the peak recorded from the sample irradiated by electron beam (Fig. 1c). Positions of other peaks coincide with those measured from the samples grown by different ways. The low temperature "tail" in this curve is because of "afterglow" of the sample containing a small amount of nitrogen. It should be mentioned that in the experiments on TSC of doped Ar solids [8] the peak at 22 K was only one observed in TSL measured in the emission of $O_2$ (Herzberg bands). It was found that O atoms start to diffuse at about 20 K to form molecular centers $O_2$ and this process of thermally stimulated atom diffusion results in recombination of neutral atoms yielding chemiluminescence. The authors of [8] observed also TSC at about 22 K in oxygen doped samples. It was explained by recombination of $O^-$ ions with neutral O atoms followed by detachment of electron from $O^-$. Activation energy estimated for the 22 K peak is $E_t = 61$ meV [9]. The binding energy $E_b$ of electron to $O^-$ is defined by electron affinity $E_a$ of oxygen atom $E_a = 1.461$ eV [22] and polarization energy $E_p$ of Ar matrix $E_p = -1.15$ eV [23]: $E_b = E_a - E_p = 2.61$ eV. Because of a big difference in $E_t$ and $E_b$ the peak at 22 K clearly cannot be attributed to a direct process of thermally activated electron detachment. The other possibility suggested [8] is a thermal diffusion of O atoms and $O^-$ ions. However, ions are characterized by short-range mobilities as it was demonstrated in the experiments on transmission of ions through rare-gas films [24]. In contrast with this neutral atoms in cryogenic solids yield long-range mobilities with migration length of 30 nm [6,25]. The high intensity of the 22 K peak in the glow curve taken from the sample deposited from the discharge is not unexpected because the conditions of growing provide the sample with high concentration of radicals and ions [13]. Also under these conditions one can expect the formation of O–O pairs with different interatomic distances. Recombination of close and widely spaced pairs is thought to be responsible for the broadening of the peak in this case.

From other side the peak at 22 K was also observed in recombination luminescence of self-trapped holes—intrinsic ionic centers $Ar_2^+$, with electrons [11,19] and under recombination of $CO^+$ guests with electrons [9]. These experimental findings demonstrate directly that electron transport is involved in relaxation processes at this temperature. Analysis of the whole set of data on TSEE and spectrally resolved TSL enables us to restore a subsequence of atomic and electronic elementary processes of relaxation in the temperature range around 22 K. O atoms generated in the matrix under irradiation and trapped in substitutional and perhaps in interstitial octahedral sites start to diffuse upon heating most likely by vacancy mechanism. Note that the estimated energy of vacancy formation near a guest O atom in solid Ar

is of $90 \pm 30$ meV, that is close to activation energy for 22 K peak $E_t = 60$ meV [9]. Recombination of O atoms results in a formation of $O_2^*$ molecule in the bound state $A^3\Sigma_u$ by the reaction

$$O(^3P) + O(^3P) \rightarrow O_2^*(A^3\Sigma_u) \rightarrow O_2(X^3\Sigma_g) + h\nu(2.1\text{–}3.6\ \text{eV}) \quad (1)$$

Relaxation to the lowest vibrational state proceeds nonradiatively via phonon mechanism. The energy release into the lattice does not exceed the Debye energy 12 meV that is much less than binding energy of electron in deep traps $E_b = 2.61$ eV for $O^-$ and $E_b = 1.6$ eV for $O_2^-$ while under radiative transition $A^3\Sigma_u \rightarrow X^3\Sigma_g$ the energy released exceeds $E_b$ for both kind of deep traps. Note, that because of the wide conduction band (several eV) this radiation can produce both "cold" electrons at the bottom of the conduction band from deep traps and "hot" electrons at the top of the conduction band released from shallow traps. Mobilized electrons can then escape from the surface or recombine with positively charged intrinsic and extrinsic centers.

In order to verify the suggested mechanism involving radiative atomic and electronic processes by the reaction (1) we performed experiments on PSEE. The experiment was carried out at $T = 7$ K to exclude all temperature assisted processes. The Ar matrix was doped with $O_2$ ($10^{-4}$). Under irradiation by electron beam $O^-$ and $O_2^-$ deep traps are inevitably formed and one can expect that the electrons can be detached from these centers by photons of appropriate energy. No electron emission from the "dark" sample was observed. The laser was tuned to 2.71 eV (the most intensive band of the Herzberg progression). A sharp rise of electron emission was observed followed by much slower decay (Fig. 2). Blocking of the laser light resulted in break off the signal. Decay of the emission continued from the same level of signal unblocking laser light. The decaying portion of the curve characterizes the decrease of the mobile electron density in the detrapping process. Neglecting retrapping the decaying portion of the emission current-time transient curve may be represented by the expression [26] $N_c = g\tau_c N_{t0} \times \exp(-gt)$, where $N_c$ is the free carrier density, $N_{t0}$ is the initial concentration of electrons in the traps, $\tau_c$ is the effective life-time of the carriers in the conduction band and $g$ is the product of the photon density and the effective electron–photon interaction cross-section. If suggest the same interaction cross-section for electrons in different traps and photons the decay is supposed to be single exponential. A first portion of the decay curve in the range not too far away from the peak can be fitted by single exponent with $\tau = g^{-1} = 60 \pm 10$ s. The deviation observed in the curve on a long time scale can be caused by retrapping of electrons. The data obtained seem to confirm suggested radiative mechanism of energy relaxation involving atomic and electronic processes and demonstrate their interconnection.

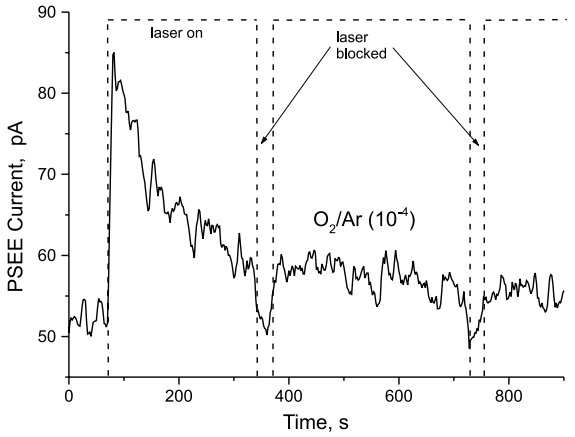


Fig. 2. Yield of PSEE from $O_2$ doped solid Ar under irradiation by laser light (2.71 eV). Current behaviour while blocking laser is also shown.

Another atomic process was found in preirradiated solid Ar-desorption induced by charge recombination. The temperature dependence of Ar pressure in the chamber, measured under heating of the sample exposed before to the electron beam shows clearly nonmonotonic behaviour (Fig. 3). An initial weak rise in the pressure was observed at $T$ about 10 K and the first pronounced peak was recorded at $T = 15$ K that is much lower then the sublimation temperature $T_{sb}$ of solid Ar ($T_{sb} = 30$ K) [14]. Then slow variations of the pressure were detected with maxima at about 20 and 35 K. The sharp decrease of the pressure at about 42 K is

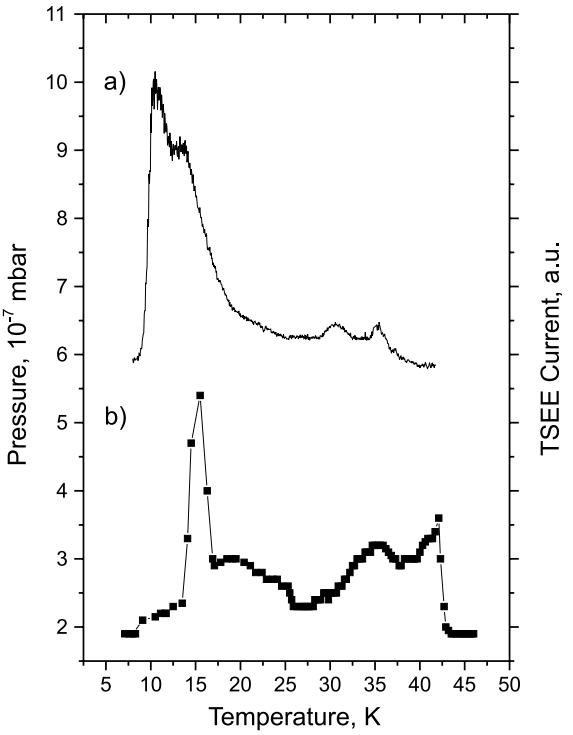


Fig. 3. TSEE current from a standard film of pure Ar gas (a) and measured simultaneously pressure in the experimental chamber (b).

more likely due to cooling of the sample surface at intensive sublimation. Comparison of the pressure curve (Fig. 3b) characterizing the yield of neutral atoms from the sample with the yield of electrons (Fig. 3a) reveals a correlation in peak position that points to a common origin of effects underlying these phenomena. Indeed, the relative intensities of the peaks in the current and pressure curves differ significantly. It should be mentioned that we observed for Ar solids macroscopic charging effect. Changing the Faraday plate polarity resulted in some suppression of the TSEE signal which was however still detectable. Accumulation of uncompensated negative space charge in the sample generated by trapped electrons makes it easy to detect currents but certainly influences the electron extraction efficiency. A decrease of the electric field in a course of TSEE causes some reduction of the extraction efficiency. Another manifestation of the space charge is a shift of first most intensive maxima to lower temperatures with increasing of the space charge. In our experiments the shift did not exceed 1 K. Electrostatic effects due to charged traps in RGS were also observed in Ref. [27]. As it was already mentioned in accordance with the TSL data the 11 K peak is related to surface traps. It is clearly seen in the disperse samples and its intensity decreases after annealing. The peak dominates in the TSEE especially at low energy of an electron beam and has pronounced intensity in the TSL measured at the wavelength of the intrinsic recombination emission [19] that is well-known M band of self-trapped excitons at 9.7 eV. On the contrary in the TSL measured in the band of extrinsic recombination emission (Cameron band of CO) this feature is fairly weak [9] because of low probability $CO^+$ recombination with electrons at the surface at low concentration of the dopant ($10^{-3}$). The second TSEE peak at 15 K correlates with the peak observed in the total yield of TSL [3] and spectrally resolved yields of TSL recorded in the bands of intrinsic [11,19] and extrinsic [9] recombination luminescence. The correlation of these peaks in TSEE and TSL clearly demonstrates the common intrinsic origin of the electronic relaxation processes involving excitation of electrons to the conduction band followed by charge transport by highly mobile electrons to intrinsic centers, extrinsic ones and to the surface. The peak at 15 K stems from levels of radiation induced defects. Formation of permanent Frenkel pairs stimulated by an excitation of electron subsystem was observed experimentally [28,29] and supported by theoretical study [30]. The most probable configuration of the defects is supposed to be the "dumb-bell" of $\langle 1\,0\,0\rangle$ configuration. Because of negative electron affinity one can expect localization of electron at the vacancy. Note that despite of the common origin processes of electron emission and recombination are competitive and therefore recombination induced desorption depends on the interplay between these relaxation paths. In all experiments variations in pressure followed the free carrier density. Energy absorbed during the irradiation and stored by localized charge carriers and reactive neutral species is released in the form of light (recombination emission) and kinetic energy of "hot" lattice atoms by the reaction

$$Ar_2^+ + e \rightarrow Ar_2^* \rightarrow Ar + Ar + \Delta E_k + h\nu\ (9.7\ eV) \quad (2)$$

There is about 1 eV energy release available for conversion to kinetic energy of lattice atoms. The process (2) as a source of energy to desorption was suggested [31] and observed experimentally [32]. Recombination induced enhancement of the total desorption yield from solid Ar was found under selective excitation above energy gap $E_g$ [33]. It is interesting that in our experiments we revealed branching of the relaxation paths even at the stage, which starts from thermalized electrons and self-trapped holes.

## 4. Summary

In this study we applied the activation spectroscopy techniques to study relaxation processes in preirradiated solid system $O_2$–Ar. The results were obtained combining the TSL method with measurements of TSEE in weak external electric fields, PSEE and desorption of neutral atoms. The study enables us to analyse the origin of electron traps in more detail and to shed light on thermally assisted electronic and atomic processes in preirradiated solids. A new effect was found—desorption of atoms from preirradiated solid Ar observed at low temperatures much below the characteristic sublimation temperature. The low temperature desorption is suggested to be caused by thermally assisted charge recombination in preirradiated solid Ar.

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