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Getters Reactants and Clean Technologies

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Abstract

Recent research in the field of gas sorption by Li-IIA alloys has improved our understanding of those processes in the gas-alloy system that influence taking practical decisions. Three features in the behavior of reactive macrobodies with a monolithic structure appeared to be significant and should lead to the replacement of a number of conventional sorption practices with new ones. These include the temporary resistance of the mentioned macrobodies to air (sorption pause), the ultra-fast decay of intermetallic phases according to the laws of corrosion (self-grinding), and the renewal of the reactive melt surface by sedimentation of reaction products with gases into the melt volume (self-cleaning). Applications of the mentioned phenomena for clean technologies are also discussed.

Keywords

Ultra-Pure Gases, Extremely High Vacuum, Getter Reactants, Rare Gases, Clean Dust-Free Technologies

1. Introduction

Alloys of Li with IIA metals named getter reactants [1] [2] possess excellent sorption properties, however, very high chemical activity of these alloys in the form of films, powders or bodies with a porous structure limits their widespread use. Mechanochemistry was also bet on, for example, in flow-type sorption reactors [3]-[5], where the starting material is simply ingots, which are introduced into the working chamber in air and then ground in the process medium at the required rate. However, this solution also has drawbacks. Among them, let us point out pressure drop and problems with the separation of the final products of the gas-metal reaction.

This critical view of mechanochemical methods has become more acute with the growing demand for pure materials and ultra-clean vacuum level production and laboratory conditions. New technological frontiers place more stringent demands on sorbents, requiring them not only to have increased productivity but also higher purity levels of the final product, with an emphasis on the complete absence of dust particles.

And here, a real surprise from Li alloys with IIA metals is the new knowledge about how they interact with gases [6]-[10], suggesting that many clean-technology problems could be solved using these reactive alloys. Their sorption behavior in gas environments stands out sharply from that of traditional sorbents such as MOFs, NEGs, and zeolites. Let us show the special properties of the given alloys and their advantages in solving clean technology problems.

2. Three Pillars of Getter Reactants

What are the main peculiarities of getter reactants as a sorbent material? At the level of empirical approach, we can talk about three features that elevate these getters over existing sorbents in solving the urgent technological problems. These are:

2.1. Loose Structure of the Cover Layer

Ingots of getter reactants, when in contact with active and low-active gases, are quickly covered with reaction products in the form of an interphase layer separating the two initial phases, gas and solid. Then the growth rate of this layer reduces by many times over a limited interval of time, called the sorption pause [6] [8]. The superiority of getter reactants over other sorbents is connected with this pause.

The described sorption scheme is based on experience and on the Pilling-Bedworth hypothesis [11] regarding the loose structure of the products of reaction of IA and IIA metals with atmospheric gases. This gas-permeable structure provides getter reactants with a sorption advantage, as it allows gases to gradually pass through to the metal body for its subsequent corrosive decomposition (see Chapter 2.2 below), whereas adsorbents or getters of NEGs type upon contact with gases are covered with a thin passivated film, which completely stops the sorption process.

2.2. Corrosive Decay of Ingots

The ability of Li alloys with IIA metals to rapidly destruct their structure under the influence of gases into individual particles [7]-[9], which then become increasingly finer, is of great practical importance. This self-grinding of the initial ingots, where gases take on the primary work of feeding the sorption process with areas of fresh metal surface, eliminates the need for the production of highly dispersed sorbents, films, powders, and bodies with a highly porous structure.

The given disintegration of the reactive macrobody is the joint result of several processes: migration of gases through the loose product layer to the alloy surface, gas diffusion into the alloy volume through microcracks and grain boundaries, chemical reactions in the body volume with the creation of internal pressure zones, and, finally, the growth of microcracks with the destruction of the ingot [12] [13]. This spontaneous emergence of more and more new areas of the sorption surface leads to an unprecedented acceleration of the overall kinetics of the sorption process.

2.3. Self-Cleaning of Liquid Getter Reactants

An exceptionally valuable property of Li alloys with IIA metals is their ability to free their surface from solid products of reactions with gases when heated to temperatures above the alloy's liquidus point [10] [14]. The liquid state of the alloy triggers a sedimentation mechanism of transferring the mentioned products from the gas/melt interface into the melt volume by gravitational forces. This renewal of the melt surface opens the door for getter reactants to many applications.

The reactive melt is fundamentally superior to solid sorbents when it comes to the purity of the operating environment from dust particles. Sedimentation cleaning of the melt surface eliminates the very possibility of accumulation of products of reactions with gases on its surface. Furthermore, the melt is capable of binding foreign solid particles by wetting them.

3. Solid-Phase Getter Reactants

Figure 1 shows the sorption curve $c(t)$ of Li–IIA metal alloys in a usual atmosphere at T_{room}, where $c(t) = \Delta m(t)/m_0$, m_0 is the initial mass of the sample in the form of a macrobody with a monolithic structure, and $\Delta m(t)$ is the mass increment over time t.

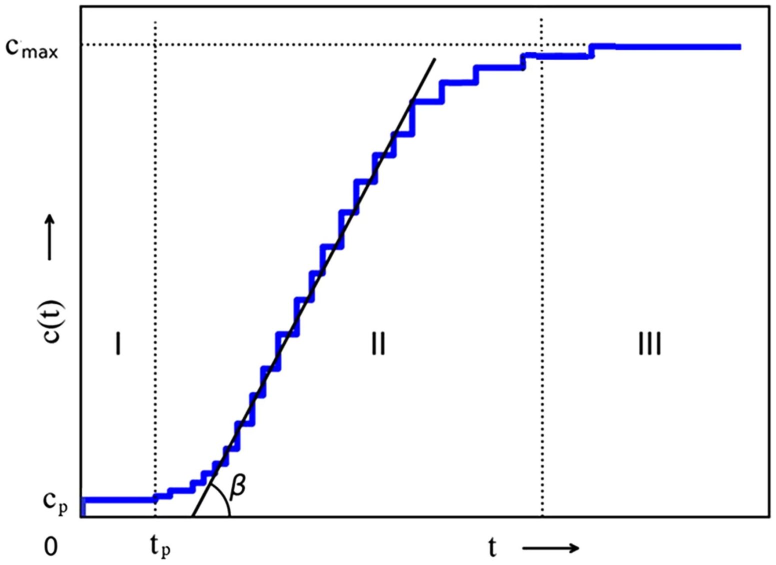


Figure 1. General view of the sorption curve of intermetallic phases in Li-IIA metal systems. I: Sorption pause; II: Stage of corrosive decomposition of the cast body, β: average slope of the $c(t)$ curve; III: Stage of attenuation of the sorption process, and $c_{\max}$: limit of sorption capacity.

Let us emphasize that here the samples are understood to be alloys with a large fraction of the intermetallic phase or consisting entirely of it. This choice is based on experience [8], which shows that the sorption of gases by intermetallic compounds of Li-IIA metal systems occurs many times faster than sorption by elemental metals [15] [16].

This behavior of reactive intermetallics is explained by their mechanical weakness and brittleness, which accelerate the corrosive decomposition of the alloy, and with it, the sorption kinetics. The new sorption mechanism of these alloys changes the usual image of the monotonic curve $c(t)$, giving it a stepped shape (**Figure 1**). In general, it can be seen that there is a certain similarity with the parabolic breakaway curves [17], however, the latter describe the destruction processes only in the layer of products on the surface, and not the disintegration of the entire getter body, as in **Figure 1**.

Let us divide according to [9] the $c(t)$ curve into three parts (**Figure 1**), where I is the initial stage, called the sorption pause, II is a series of steps indicating the corrosive decomposition of the ingot and intensive gas capture, and III is the stage of attenuation of the sorption process with the exhaustion of the getter mass at the $c_{\max}$ level. This approach to analyzing the sorption process is pragmatic, highlighting those of its features that are of interest for applications.

Sorption pause I on the graph looks like a vertical segment with ends $(0,0)$ and $(0,c_p)$, which then continues as a horizontal segment with ends $(0,c_p)$ and (t_p,c_p). This pause provides applications with an ideal chance for open installation of the getter reactant at its working place, since, according to available data, the point t_p falls within the range of 10 - 15 minutes to several hours [8], which satisfies any time requirements. Moreover, at stage I the thickness of the sacrificial layer is small and therefore, c_p is also small. The share of getter material lost during assembly in air can be reduced by increasing the average size of the getter body [5] [7].

Stage II, otherwise known as self-grinding of reactive intermetallic compounds, makes them unattainable for other sorbents in terms of specific sorption capacity $c_{\max}$, as shown in [8]. In its turn, sorption kinetics, determined by the slope β (**Figure 1**) can be preset. Factors regulating sorption kinetics include the getter composition (**Figure 2**), its microstructure (**Figure 3**), and the value of the initial specific surface area of the getter (**Figure 4**).

So, two phenomena, the sorption pause I and the corrosion self-grinding II, contribute new insights into sorption processes, correcting common understanding in this area and confirming the practical value of reactive alloys. Thus, a loose cover layer, typically perceived negatively, proves beneficial here, minimizing getter loss during installation in air but allowing gases to pass to the boundary with the alloy, creating conditions for its subsequent disintegration [8]. Corrosive decay, a clear scourge for most metal products and constructions, serves as the main driving force of the sorption process for getter reactants [6] [8].

Let's move on to the potential applications of solid-phase getter reactants and demonstrate their strengths in solving pressing problems in resource efficient and clean technologies.

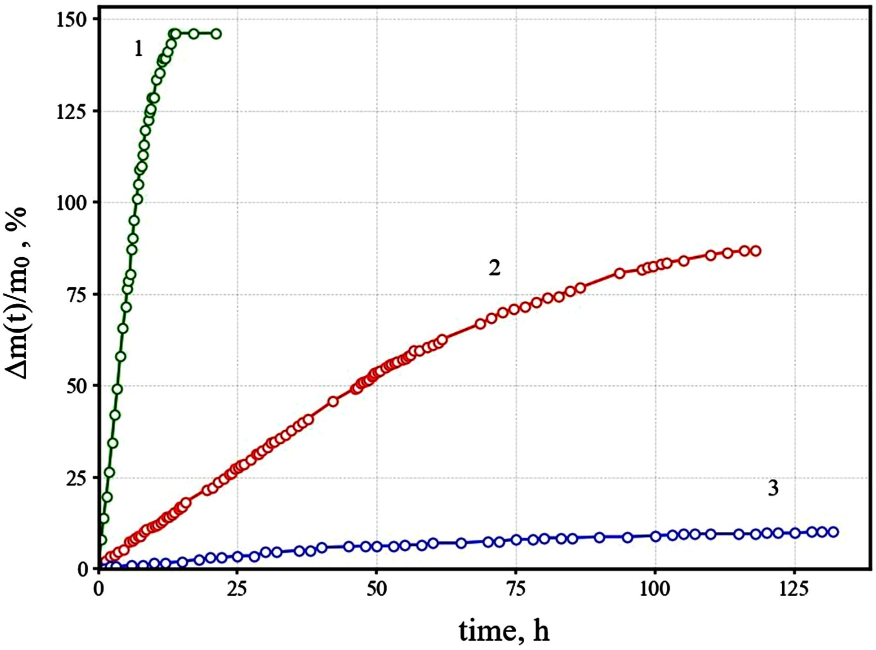
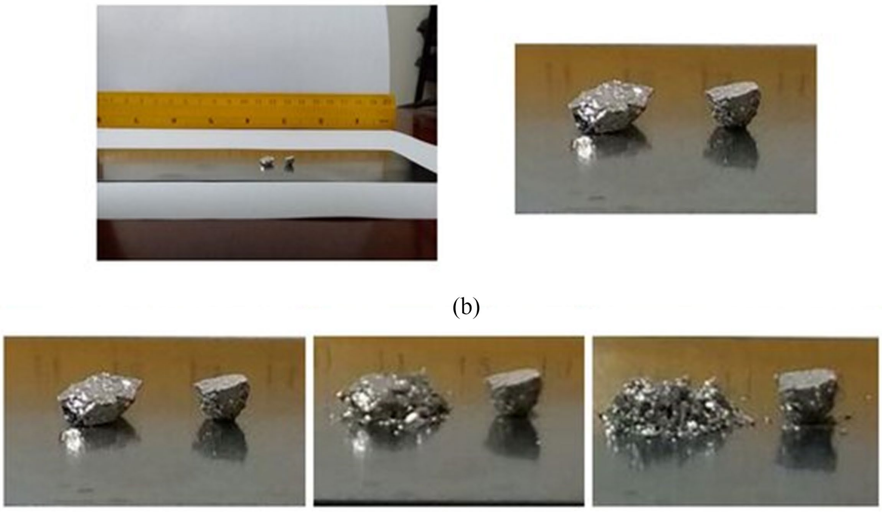


Figure 2. Sorption curves of reactive intermetallic compounds under normal atmospheric conditions at T_{room} [7]. Here, curve 1 describes the phase of composition $Li_{23}Sr_6$, curve 2—the phase of composition $CaLi_2$, and curve 3—the phase of composition $CaMg_2$.

(a)



(b)

t = 0, h
t = 3, h
t = 22, h

Figure 3. Decomposition of two samples of the composition $Ca_{0.35}Li_{0.45}Mg_{0.20}$ in standard conditions [9]. (a) Initial state of the samples and a ruler for estimating their size; (b) Decay process over time (hours). The sample on the left was taken from the bottom of the growth crucible; the sample on the right, from its central part.

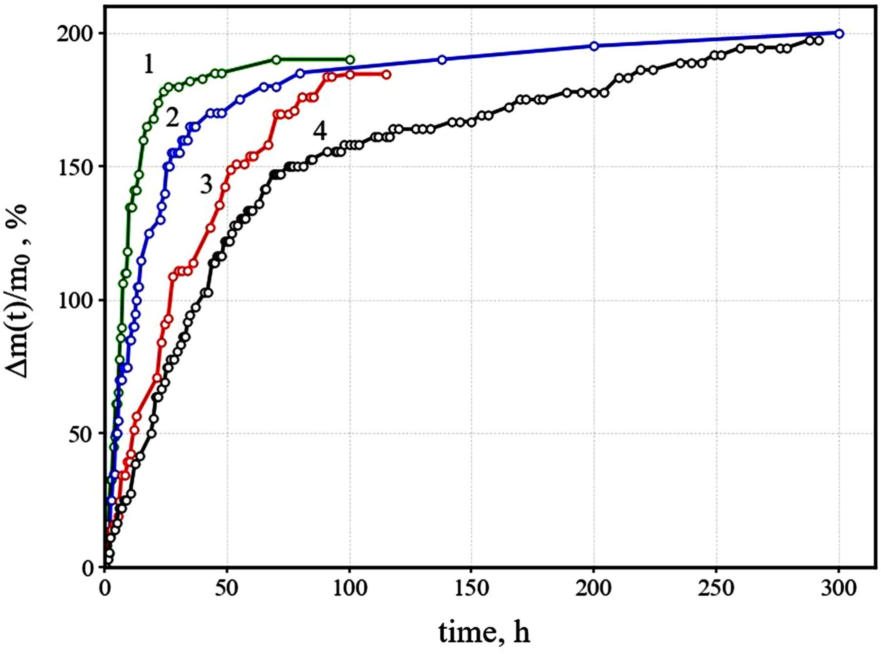


Figure 4. Sorption curves of the Laves phase $Ca_{0.33}Li_{0.48}Mg_{0.19}$. Here, curves 1, 2, and 4 [8] are supplemented by curve 3, creating a convincing picture of the influence of the initial values of the specific surface area of the getter material on its sorption kinetics in air. These values in the given case are 71 cm^2/g (curve 1), 39 cm^2/g (curve 2), 30 cm^2/g (curve 3), and 22 cm^2/g (curve 4).

3.1. Single-Act Mechanical Activation

As mentioned above (see **Figure 4**), the sorption kinetics of an alloy depends on its specific surface area, which suggests a simple solution for increasing these kinetics by single-act destruction of ingots in the target medium, avoiding losses during the assembly stage, etc.

Technically, this method comes down to using a hermetically sealed vessel, divided into two parts by a partition permeable to gas molecules. The first part of this vessel is the target chamber with a pure medium or medium to be purified, while the second is a reactor with a getter and a reserve space for solid waste from the reaction of the getter with the impurity, which enters from the first part through the partition.

The reactor serves not only as a place for crushing the getter alloy and capturing the impurity but also as a place for securely retaining the solid waste thanks to the partition. This design is suitable for purifying cryogenic gases during storage in a gas pool, which effectively eliminates the time spent on the sorption process. The same single-act grinding concept can also be used in products such as semiconductor packaging, etc.

3.2. A New Concept of a Vacuum Window

Development of the vacuum window, which began about 30 years ago, led to the first commercial product [18], which consists of glass panels joined by hermetically sealing at the edges with a sealing material and having an evacuated gap. These windows contain a getter in the gap, and one of the panels has a hole for pumping down the air.

This new mass-market product is designed to dramatically reduce energy losses in office and residential buildings [19]. However, the 20-year service guarantee announced by the manufacturers is approximately half that was previously promised, and the price of this product is very high. It is also worth noting that despite intensive advertising of the new windows, their key component, the getter, has remained largely unnoticed, although it is responsible for the window's functionality [2].

Considering that the production costs of any sorbent for a vacuum window are negligible compared to the cost of producing the window itself, and that getter reactants surpass all other sorbents by orders of magnitude in specific sorption capacity, we come to the conclusion that the future of vacuum glazing and getter reactants are inseparable. Based on this, the first model of a vacuum window with getter reactants was proposed in 2016 [20], along with its rationale [2].

The getters in this model were alloys of Li, Na, Mg, Ca, Sr, and Ba , which, after grinding, were introduced under vacuum into getter channels located along the window edge. This allowed a large getter mass to be loaded into the window, concealing it under the frame. However, transporting the getter particles into the window under vacuum complicated the assembly process, which slowed the innovation.

A second model was not long in coming [6] [9]: air tests with ingots of getter reactants demonstrated their tolerance to atmospheric gases and the possibility of their open-air installation in a window. Now, nothing prevents the replacement of NEGs in a vacuum window with getter reactants, exploiting all the advantages of reactive alloys, leading to cost reduction, a significant increase in window service life, and an expanded range of window products [9].

This list includes not only the already well-known vacuum window design, but also a simplified version of an energy-saving vacuum window, the production of which does not require a vacuum pump. The third, final model, currently under development, is constructed using a single-act crushing of getter macrobodies in a reactor channel located along the lower edge of the window. This channel is separated from the vacuum gap by a gas-permeable partition [8], and the getter is introduced into the channel openly (in air) and crushed after sealing.

4. Liquid-Phase Getter Reactants

A true breakthrough in the field of ultraclean vacuum and ultrapure inert gas environments is becoming possible with the simple step of converting getter reactants into a liquid state (see Chapter 2.3). Changing the phase state of the getter material results in the emergence of new mechanisms for influencing the sorption process in Li-IIA metal alloys [10].

The liquid state of the alloy provides a simple and reliable way to control the reactant surface area, and therefore the sorption kinetics. Furthermore, melting the getter initiates its self-cleaning mechanism, where solid reaction products are released from the melt surface into its volume under the influence of gravity. Thus, the melt surface is cleaned, ridding the environment from free solid particles and maintaining a high specific rate of gas pumping.

This behavior of reactive melts should be of interest in cases where traditional sorbents are used, which become sources of dust for the environment as they wear out. Furthermore, the liquid reactant helps achieve a maximum vacuum level of $\sim 10^{-14}$ mbar [10] [14], which is two orders of magnitude higher than currently available level.

Like solid-phase getter reactants, melts have their own criteria for selecting the optimal composition. While the advantages of the former are based on the brittleness of intermetallic phases in Li-IIA metals systems, the latter are the more successful in cleaning the medium the lower their liquidus temperature. Low operating temperatures of the melt keep its components in the condensed state, which is in the interest of a clean environment.

Eutectic alloys based on Li in the Li-Ca, Li-Sr, Li-Ba, Li-Ca-Sr, Li-Ca-Ba, Li-Sr-Ba, Li-Ca-Sr-Ba systems, and the eutectic Li-10 at% Ce [21] meet these conditions. If we limit the operating range of the melt to $150^\circ\text{C} - 200^\circ\text{C}$, we come to a family of reactive alloys with Li with concentrations ranging from 90 to 80 at% and below, for which vacuum of 10^{-10} - 10^{-11} mbar is achievable in accordance with the data of [22].

Reactive melts are easily adapted to any sorption application, as they require only one thing for success: to be in contact with the process medium. Applications for this product are seen not only in nuclear energy or particle accelerators, but also in many other areas requiring pure materials and a clean operating environment. Let us consider two such examples, one from the field of scientific instruments and the other from the field of production.

4.1. Analysis and Visualization of Chemical Reactions

In recent years, liquid metals have become the subject of intensive research using electron and optical microscopy, as well as X-ray absorption spectroscopy, with particular attention from chemical engineering and catalysis specialists [23]-[29]. Melts of Li-IIA metals are a potential participant in this field, as direct reactions of these alloys with gases are more efficient than catalytic reactions. There are also other reasons for the interest in Li-IIA metal melts from the standpoint of chemistry and scientific instrumentation.

Firstly, the melts of getter reactants provide this field of chemistry and chemical process analysis with an excellent vacuum pump with an unprecedentedly high vacuum level, which captures not only all active and low-active gases, but also vapors of such elements as Cd, Zn, Hg, Tl, Pb, As, Sb, S, Te, etc. [6]. Most importantly, this pump also removes dust particles from the internal environment of measuring equipment, such as a microscope, and, due to the fluidity of the melt, allows for wide and rapid changes in gas pumping rate (**Figure 5**).

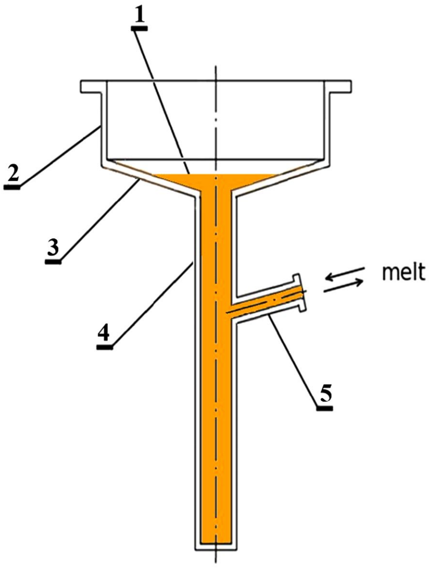


Figure 5. Getter pump with controlled pumping rate. 1: melt surface, 2: upper cylinder (expansion chamber), 3: cone, 4: lower (base) cylinder, 5: port leading to the control tank with the melt (not shown); the pumping rate is minimal when the melt surface 1 decreases to the level of cylinder 4, but increases as it rises, increasing by a factor of $(R/r)^2$ at the level of cylinder 2 (here R is the radius of cylinder 2 and r is the radius of cylinder 4).

Secondly, these melts expand the technological capabilities of chemical engineering in many sorption tasks; and for microscopy and spectroscopy they offer long-lasting samples, when a single sample can replace dozens of conventional ones. The main trump card here remains the same: it is the renewal of the sample surface during melting. After measurements, the solid sample can be cleared from the products on the surface by melting, and then cooled again to its original temperature, but with a renewed surface, and all this, on the sample stage.

Getter reactants can also aid in the study of structural changes in melts during the step-by-step stages of their interaction with different substances, including metals. Such promising materials as Zintl phases [30]-[32] can also be involved in such experiments. For example, these phases are formed when liquid eutectics of $\text{Li-10 at\% Ce}$ or $\text{Li-12 at\% Sr}$ capture vapors of $\text{Cd, Zn, Hg, Pb, Te}$, and many other elements, which, when dissolved in these eutectics, form clusters with their components based on ionic-covalent bonds. These are the questions that currently interest the chemical community [28].

4.2. Production of Ultra-Pure Noble Gases

Noble gases, like vacuum, are the foundation of clean technologies. These gases are not only chemically inert, but also possess unique physical properties, without which modern industries such as lighting [33], protective atmospheres [34]-[37], medicine [38], thermal insulation [39] [40], research in the field of elementary particles [41]-[43], and so on could not exist. However, with the exception of Ar, these gases are considered rare gases, the extraction and purification of which are very expensive [44], so the development of new and more efficient methods for their processing remains relevant.

Destruction of reactive ingots by single-act crushing in a reactor chamber separated from the target environment by a gas-permeable partition is a step toward cleaner gas production. However, radical progress in this field will occur with the transition from solid reactants to their melts [10] [14], and this is not a step, but a true leap from conventional gas/solid systems to ternary gas/melt/solid systems; in the latter their own forces are acting, expanding our capabilities in the field of sorption practices.

As discussed above, the surface of the reactive melt chemically captures gaseous impurities and dust particles from the environment in contact, which then go into the volume of the melt via sedimentation mechanism, cleaning the melt surface and maintaining its sorption activity. In the known gas purification methods there are no analogues to this productive and, we emphasize, dust-free behavior of getter materials.

A fundamental advantage of getter melts is their fluidity, which simplifies the task of maximizing the specific surface area of the sorbent. The liquid reactant easily changes its surface area, conforming to the shape of the vessel ([14], see also **Figure 5**); it is capable of enhancing sorption kinetics also due to convective flows of the melt at the gas/melt interface; its production costs are low, and its chemical activity is unmatched.

Another argument in favor of the new technology is its extreme simplicity, as demonstrated by the example of a bath containing a getter reactant melt, over which there is the gas being purified (**Figure 6**).

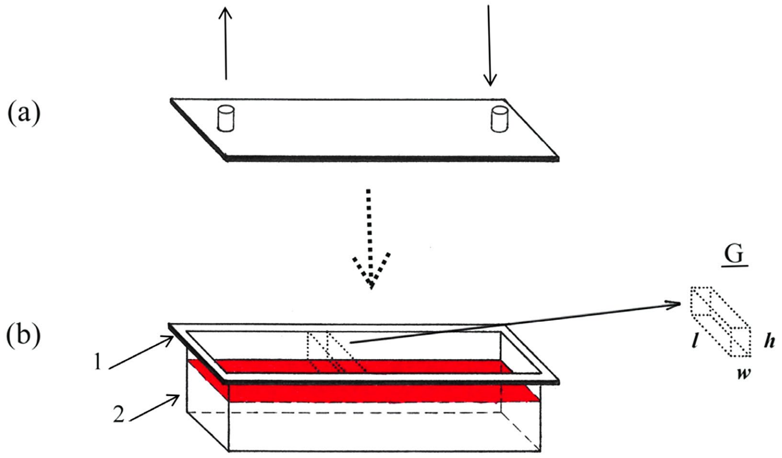


Figure 6. Sorption bath for purifying rare gases. (a) SS bath lid with gas inlet/outlet openings; (b) SS bath with melt and space for gas of height h; 1: CF flange for hermetic connection with the bath lid; 2: the body of the bath; G is the gas column of volume $V = s \times h$, where $s = l \times w$; the melt surface is marked as red. Two modes are possible: a flow mode and a static mode.

The gas pressure is slightly higher than atmospheric, eliminating the risk of gas leakage from outside. There are no restrictions on the initial impurity level in the processed gas, allowing this solution to cover both the production of ultrapure gases and the recycling of rare gases. The lid over the bath also provides a convenient location for a set of sensors and instruments that monitor and control the sorption process.

Sorption baths of this type are easily configured for both laboratory and production settings. Preliminary information on the record-breaking gas purification kinetics in such baths with a liquid reactant is contained in Appendix. By manipulating such parameters as the column height h (**Figure 6**) and the gas flow rate above the melt, it is possible to control the production of noble gases with zero active and low-active impurities, as well as the recycling of waste noble gases.

5. Conclusions

Getter reactants are a special class of chemisorbents based on Li-IIA metal alloys. They are versatile, easily adapting to any vacuum or gas application; in both their phase states, solid and liquid, they outperform existing sorbents on performance parameters; they are also a clear pillar of developing clean technologies. So, the essence of this review:

1) Solid-phase getter reactants are arbitrary-shaped macrobodies with a monolithic structure, belonging in composition to the intermetallic phases of the Li-IIA metal systems. Due to their structure, they withstand assembly in air, and at the same time during the operating stage, due to their brittleness and mechanical weakness, they quickly reach the theoretical limit of sorption capacity in a gas environment in the process of corrosive decomposition. Sorption pause and self-grinding in gas environment make these getters leaders in sorption efficiency.

2) Liquid-phase getter reactants are melts of eutectic and hypereutectic lithium alloys with Ca, Sr, and Ba at temperatures of 150˚C - 200˚C, corresponding to concentrations of $90 \text{ to } 80 \text{ at\%}$ Li or even less. A getter melt solves the main problem of clean technologies by ridding them of free solid particles. Purity level is increased by sedimentation of solid particles within the melt, and sorption kinetics becomes controllable due to the melt's fluidity, allowing for easy modification of its surface area.

3) Contribution to clean technologies. Getter reactants win in the fight against dust particles: the melt surface captures not only active gases and the products of reaction with them but also foreign solid particles. All particles with a density higher than the melt density, go into its volume under the influence of gravity forces. Further, two getter pumps are capable of creating an extremely high vacuum of $\sim 10^{-14}$ mbar, while a sorption bath with a getter melt (see **Appendix**) under appropriate conditions ensures complete removal of all active and low-activity impurity in a fraction of a second.

4) Contribution to productivity. The simple design of the equipment, the single-act mechanical activation in the solid state, and a one-stage controlled sorp-

tion process in the liquid state, the unlimited availability of consumable metals, the alloys of which in the form of primary ingots serve as the initial gas sorbent with record-breaking performance characteristics—all these are constituent parts of a new sorption technology capable of replacing a number of traditional practices. Getter reactants mobilize natural phenomena, manifesting themselves in the form of a beneficial sorption pause, self-grinding, and self-cleaning, increasing productivity and reducing costs in sorption technologies.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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Appendix

Let us single out a gas column in the form of a rectangular parallelepiped G of height h and bases of area s above the melt in the bath (**Figure 6**), where the lower base serves as the boundary with the melt, and the upper base, with the bath lid. The number of impacts of gas molecules on the melt surface s during a time t is denoted by $n = vst$, where $v = p/\sqrt{2\pi mkT}$ is the number of impacts per second per $1\,\text{cm}^2$ of melt surface, p is the gas pressure above the melt, m is the average mass of gas molecules, k is Boltzmann's constant, and T is the bath temperature. Let us calculate the time t_c at which the number n equals the number of gas molecules in the above-mentioned column G of volume $V = sh$.

Using the equation of state for gases $pV = NkT$, where N is the number of gas molecules within a volume V, we find that $t_c = \alpha h$, where $\alpha = \sqrt{2\pi m/kT}$. Assuming the following values: $p = 1.1\,\text{bar}$, $h = 1\,\text{cm}$, average molecular mass $m = 40\,\text{g/mol}$, and $T = 450\,\text{K}$, we arrive at $t_c = 8\times10^{-5}$ sec. Collision of any particle, except for inert gas atoms, with the reactive melt leads to its capture, therefore, the value of t_c under certain conditions can provide an estimate of the time required for complete purification of the initial noble gas from active and low-active impurities.

Indeed, in terms of impurity level, rare gases can be divided into two sorts: gases, purification of which involves a sedimentation process, and gases subjected to instantaneous purification. The dividing line here is the concentration at which the amount of impurity in the initial gas column above the melt is sufficient to form a monolayer of reaction products on the melt surface. Let's estimate the value of this concentration.

Elementary calculations performed for the cases $h = 0.1$ cm, $h = 1$ cm, and $h = 10$ cm show that the desired concentration is 99.95%, 99.995%, and 99.9995% of the noble gas, respectively. In each of these three cases, these gases release all their active impurity to the melt within a time of $t_c \sim 10^{-4}$ sec. In terms of both the time scale and the level of purity of the final product, with the complete removal of all active gases and dust particles, this is a remarkable result.