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DEVELOPMENT OF REACTION CENTERS WITH THERMAL DECOMPOSITION OF ORTHORHOMBIC AMMONIUM PERCHLORATE AND THE ROLE OF DISLOCATIONS IN THIS PROCESS

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Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

FOLLOWING ARE THE CORRESPONDING RUSSIAN AND ENGLISH

DESIGNATIONS OF THE TRIGONOMETRIC FUNCTIONS

Russian	English
sin	sin
cos	cos
tg	tan
ctg	cot
sec	sec
cosec	csc
sh	sinh
ch	cosh
th	tanh
cth	coth
sch	sech
csch	csch
arc sin	$\sin^{-1}$
arc cos	$\cos^{-1}$
arc tg	$\tan^{-1}$
arc ctg	$\cot^{-1}$
arc sec	$\sec^{-1}$
arc cosec	$\csc^{-1}$
arc sh	$\sinh^{-1}$
arc ch	$\cosh^{-1}$
arc th	$\tanh^{-1}$
arc cth	$\coth^{-1}$
arc sch	sech^{-1}
arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

GREEK ALPHABET

Alpha	Α	α	α		Nu	Ν	ν	
Beta	Β	β			Xi	Ξ	ξ	
Gamma	Γ	γ			Omicron	Ο	ο	
Delta	Δ	δ			Pi	Π	π	
Epsilon	Ε	ε	ε		Rho	Ρ	ρ	ρ
Zeta	Ζ	ζ			Sigma	Σ	σ	ς
Eta	Η	η			Tau	Τ	τ	
Theta	Θ	θ	ϑ		Upsilon	Τ	υ	
Iota	Ι	ι			Phi	Φ	φ	ϕ
Kappa	Κ	κ	κ	κ	Chi	Χ	χ	
Lambda	Λ	λ			Psi	Ψ	ψ	
Mu	Μ	μ			Omega	Ω	ω	

DEVELOPMENT OF REACTION CENTERS WITH THERMAL DECOMPOSITION OF ORTHORHOMBIC AMMONIUM PERCHLORATE AND THE ROLE OF DISLOCATIONS IN THIS PROCESS

A. V. Rayevskiy, G. B. Manelis

Studied in works [1, 2] was the topography of the decomposition of separate crystals of orthorhombic NH_4ClO_4 directly in the course of the reaction, the assumption is discussed about the participation of the dislocations in this process, and preliminary research is conducted on dislocations in crystals of ammonium perchlorate. Recently in literature there again has appeared a vast amount of works [3, 4] devoted to the research on the topography of decomposition of NH_4ClO_4. The interest in this question is clear, since, on the one hand, all the proposed models of the decomposition of NH_4ClO_4 must explain the topography of decomposition, and, on the other hand, the data obtained from microscopic observations can substantially aid in the creation of a new model. In work [3] it was indicated that, unlike our results [1], the movements of the nuclei in the zone of reaction was not observed. However, as it seems to us, the movement of the nuclei is a very important feature in the development of the reaction of the decomposition of the orthorhombic crystals of NH_4ClO_4. In work [4] the features of topography are explained by the internal cracking of the crystal, the movement of the nuclei is contradicted, and their rapid rearrangement is discussed; the role of the dislocations is not examined.

In connection with this we conducted a more detailed research on the topography of the decomposition of NH_4ClO_4 crystals, during which special attention was given to features of the development of reaction centers (zonal nature of the course of reaction, the movement and the multiplication of the nuclei in the zone of reaction) and, in connection with this, the role of the dislocations in this process. In this report the basic results of the performed work are briefly set forth.

Crystals of NH_4ClO_4 grew according to the method described in work [1]. Decomposition was carried out under isothermal conditions (temperature range, 196-235°C). All the observations and photographing were conducted with the aid of a microscope directly in the course of the reactions. In the necessary cases stereomicrophotography was used. Etching was implemented in a vacuum heating stand because of sublimation directly in the course of the reaction. Kinetic measurements were conducted in air on the microbalance ATV-3 [5]. It was noticed that during the induction period some crystals became turbid unevenly. The turbid sections consisted of a large number of immobile semi-transparent spheres (diameter, <1 μm) located by chains along the direction [310] of the crystal. The crystal surface either remained unchanged or was etched because of such sublimation. The induction period was finished as soon as centers of the decomposition began to appear. They were formed both near the surface (at a depth of 10-30 μm) and in the space of the crystal (Fig. 1). The developed centers had the form of ellipsoids of revolution and consisted of stationary nuclei surrounded by the zone of reaction in which the movement and multiplication of the nuclei with a diameter of <2 μm occurred. This nature of the development of reaction leads to assumption about

Figure 1. Stereophotograph of a NH_4ClO_4 crystal in the process of decomposition (dark field).

the participation in it of dislocations - immobile ones which were formed with the crystal growth (during the induction period), and - moving and decomposing ones in the zone of reaction (with an increase in the centers of decomposition).

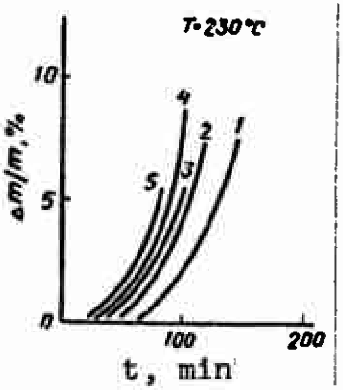

Figure 2. Dependence of the degree of decomposition on the time of the NH_4ClO_4 crystals with a different average dislocation density: 1 - ~10^2 cm^{-2}; 2 - 10^3 cm^{-2}; 3 - 10^4 cm^{-2}; 4 - 10^5 cm^{-2}; 5 - 10^5 cm^{-2}.

For an explanation of the role of dislocations additional experiments were carried out. Thus the decomposition of the crystals of identical habit and weight, after preliminary etching for determining the average dislocation density, was investigated with the aid of thermobalances. Curves of the dependence of the degree of decomposition on time at a different average dislocation density are given on Fig. 2. However, these results should be examined only qualitatively. The section of the crystal in the region of the developed center was etched by sublimation (Fig. 3). One should note the conformity of the pits of etching to the nuclei and their increased concentration near the center of the decomposition. The movement of the nuclei in the zone of reaction with an increase in the center of the decomposition is shown on Fig. 4a and b (microphotographs with exposures: a - 2; b - 90 s). The tracks which could be formed only in the case of the movement of the nuclei are clearly visible. By the direct calculation of the density of the nuclei in the zone of reaction (on the photographs) there was determined the temperature dependence of the width of the zone in direction [010] with the simultaneous measurement of the rate of propagation of the zone (rate of growth of the centers). While the activation energy for the width of the zone was changed from one center to the next and lay in the interval of 5-15 kcal/mole (on the average, about 10 kcal/mole), the activation energy of the rate of movement of the zone remained

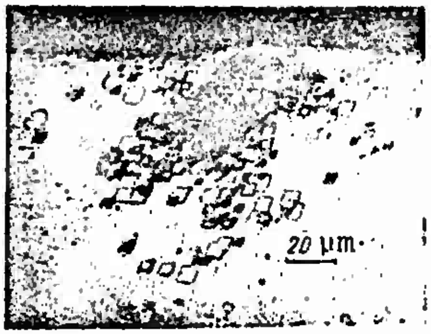

Figure 3

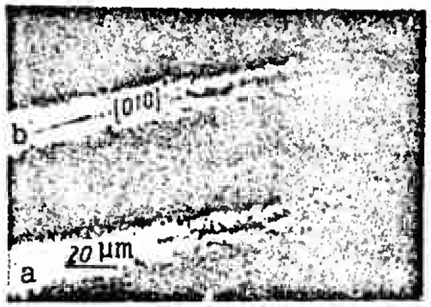

Figure 4

Figure 3. Photograph of a section of the crystal with a developed center of decomposition (reflected light).

Figure 4. Microphotograph of the center of decomposition.

constant for all centers and equal to about 30 kcal/mole. Figure 5 shows the change in density of the nuclei in the width of the zone for the same center (photographing of the density of the nuclei was produced after the establishment of the constant rate of growth of the center at the assigned temperature). The possibility of the appearance of new dislocations under the action of mechanical stresses, which are formed because of the accumulation of decomposition products in the crystal, was checked in the following experiment. The crystals were irradiated by a probe of X-rays 0.2 mm in diameter, and then they were subjected to heating up to 160°C. After the development of the reaction in the radiation zone (but before the appearance of the usual centers of decomposition) the latter was investigated under a microscope. Figure 6 gives a stereophotograph of the irradiated zone with chains of dislocations cambered in the direction [010]. By dissolution of the eutectic NH_4ClO_4-$LiClO_4$ of the section of the crystal which contains a small number of nuclei and a comparison of the spaces of the nuclei and the gas bubble formed in the solvent, the pressure of the gaseous products in the nucleus appearing in the course of reaction is determined. It proved to be equal to about 20 kgf/cm^2 at 225°C.

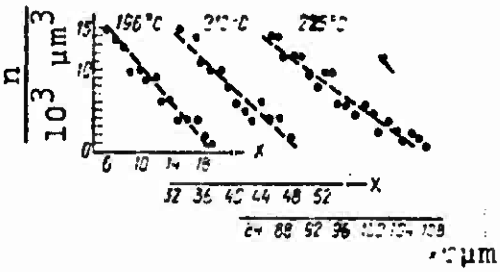

Figure 5

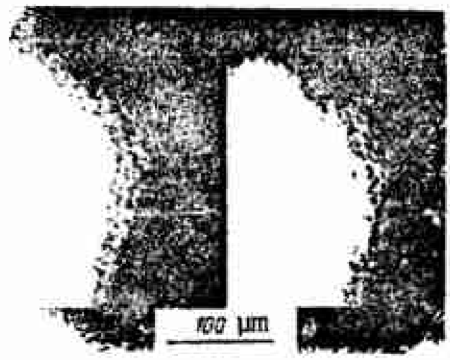

Figure 6

Figure 5. Change in the density of the nuclei in the zone of the development of the reaction of NH_4ClO_4 decompositions at different temperatures.

Figure 6. Stereophotograph of the section of the crystal irradiated by the X-ray probe.

Taking into account the facts given above, it is possible to describe the development of the centers of reaction in the following manner. Because of the facilitation of the reaction on the dislocations, the process occurs both on the stationary dislocations (induction period) and on those which are moving (period of acceleration, development of reaction centers). A growth in the centers occurs where created are conditions favorable for the movement and multiplication of dislocations under the action of mechanical stresses because of the accumulation of reaction products occurring in the extended zone. It is possible to assume that one of the reasons for the stop of the growth in the nuclei is the braking of the reaction with the accumulation of products in the nucleus under high pressure (reaction in the "closed volume" [6]). Movement of the dislocation can facilitate the transfer of products from the zone of reaction.

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