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A STUDY OF PRE-IGNITION PROCESSES IN ZIRCONIUM, TITANIUM  
AND ZIRCONIUM HYDRIDE

B. M. Ziobinskii, et al

Foreign Technology Division  
Wright-Patterson Air Force Base, Ohio

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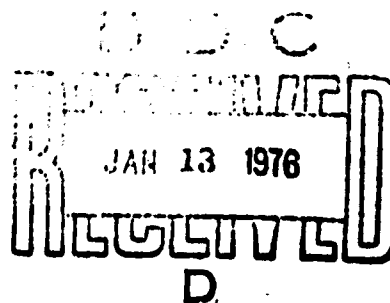
## FOREIGN TECHNOLOGY DIVISION



A STUDY OF PRE-IGNITION PROCESSES IN ZIRCONIUM, TITANIUM  
AND ZIRCONIUM HYDRIDE

by

B. M. Zlobinskiy, V. M. Mal'tsev, V. V. Kurylev




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

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

GREEK ALPHABET

Alpha	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	•	Rho	Ρ ρ ϱ
Zeta	Ζ ζ		Sigma	Σ σ ς
Eta	Η η		Tau	Τ τ
Theta	Θ θ	•	Upsilon	Υ υ
Iota	Ι ι		Phi	Φ φ
Kappa	Κ κ	•	Chi	Χ χ
Lambda	Λ λ		Psi	Ψ ψ
Mu	Μ μ		Omega	Ω ω

## RUSSIAN AND ENGLISH 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	$\operatorname{sech}^{-1}$
arc csch	$\operatorname{csch}^{-1}$
—	
rot	curl
lg	log

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A STUDY OF PRE-IGNITION PROCESSES IN ZIRCONIUM, TITANIUM  
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B. M. Zlobinskiy, V. M. Mal'tsev, V. V. Kurylev

Moscow Institute of Steel and Alloys. Department for measures  
for protection of labor.

The study of the pre-ignition processes arose from the need to more completely explain the mechanism of ignition and combustion of metals. Experiments were conducted on particles of electrodeposited zirconium (99.96% Zr), magnesium-thermal titanium (94.6% Ti) and zirconium hydride ( $ZrH_2$ )\*. [Footnote: for 1 g of metal, 240 cm<sup>3</sup> of hydrogen, less than 0.2 cm<sup>3</sup> of oxygen.]

For evaluating the growth of oxide films on metal particles (with a size from 100 to 520  $\mu m$ ) under conditions of dynamic heating microphotography was used. A graphite plate, 8 X 18 X 5 mm, was used as a heating base. Heating was accomplished with an electric current from an alternating voltage network which was supplied to the plate through a universal power-supply unit. The heating rate was regulated within the limits of 20-90 deg/s. The temperature of the particles and the heating rate were registered using a C-A thermocouple in the graphite heater. The behavior of the particles during heating (change of shape or surface structure) was recorded using the movie camera RFK-5 and microscope MBS-2. For determining the temperature of a particle two particles with different melting temperatures were transferred to each graphite plate (size, 300 and 500  $\mu m$ ). Heating of the graphite plate and filming were begun simultaneously. The moment of melting was recorded on film (at this time the particle took on an almost spherical shape). The melting temperature of the particles was determined on the basis of the rate of heating, the rate of film advance and the final temperature of the heater.

During the development of the method (on the basis of several dozen tests) a calibration graph was constructed and the reliability

of temperature determination was checked. The absolute error was  $\pm 8\%$ .

The experiment was conducted in the following manner. A small quantity of metallic powder of a determined fraction was sprinkled on a slide and using microscope MBS-1 the size and shape of the particles was estimated. The particles were measured in two mutually perpendicular directions and the mean arithmetic value was taken as the value of the average diameter. The accuracy of determination of particle size using the microscope was  $\pm 5 \mu\text{m}$ .

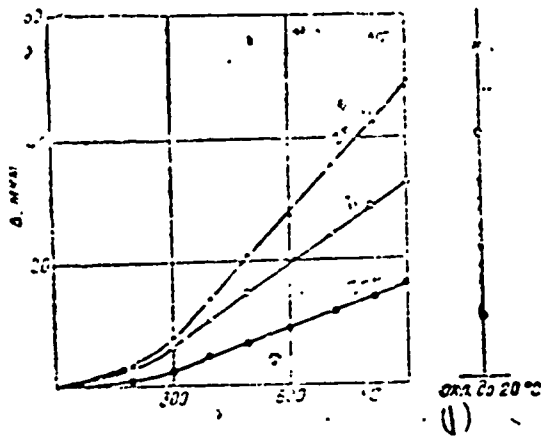
The particles selected under the microscope (those close to spherical in shape) were transferred to the center of the graphite plate using a special needle. During the experiment the heating of the graphite plate and the running of the camera RFK-5 were begun simultaneously. The particles were heated to  $900^\circ$ . After the experiment the particles which had cooled to  $20^\circ$  were once again photographed. Visual observations were also made of the behavior of the particles during heating. (through the microscope).

The films were processed on a small slide projector DM-2 (overall magnification 290-300 times). The particles were measured in two mutually perpendicular directions and their average size was determined. The thickness of the oxide film on the metal particles was estimated on the basis of the difference in the size of the oxidized particles and the initial particles taking into account thermal expansion. The maximum error during measurement of the thickness of the oxide film was  $\pm 1.33 \mu\text{m}$ .

The figure shows the dependence of the growth of the oxide film  $\Delta$  on particles of zirconium, titanium and zirconium hydride on the heating temperature ( $\Delta$  is the actual increase in the diameter of the particles). There are many similarities in the behavior of particles of zirconium and titanium during heating: they lack a clear surface of separation between metal and oxide, i.e., oxidation may continue even if the particle is covered with



a layer of solid oxide. With an increase in the temperature from 20 to 200° the increase in the average diameter of the particles is insignificant. Apparently at these temperatures the dissolution of oxygen can be disregarded [1].



The dependence of the growth of an oxide film  $\Delta$  on metal particles on the heating temperature. Diameter of the particles 200  $\mu\text{m}$ , rate of heating 30 deg/s. (1). cooling to 20°C

The beginning of heterogeneous reaction is assumed to be in the temperature interval from 200-300° for zirconium and from 300-380 for titanium. (At a temperature of 200° for zirconium and 300° for titanium the particles change color and become blue-black, and at a temperature of 300° for zirconium and 380° for titanium they begin to acquire a red color). This process is slow surface combustion. The oxide film which was formed as a result of this process covered the remaining part of the metal and prevented transition of the heterogeneous reaction into intense combustion. With an increase in the temperature (from 300° for zirconium and 380° for titanium) to 900° intense growth of the oxide film takes place on the particles as a result of the heterogeneous reaction (zirconium 100  $\mu\text{m}$ - $\Delta$  =  $\Delta'$  -  $\delta$  = 35.1  $\mu\text{m}$ , where  $\delta$  is the calculated growth of the particle due to thermal expansion,  $\Delta'$  is the average absolute increase in the diameter of the particles: 200  $\mu\text{m}$ - $\Delta$  = 48.7  $\mu\text{m}$ ; 300  $\mu\text{m}$ - $\Delta$  = 52.4  $\mu\text{m}$ ; titanium 100  $\mu\text{m}$ - $\Delta$  = 20.6  $\mu\text{m}$ ; 200  $\mu\text{m}$ - $\Delta$  = 33.1  $\mu\text{m}$ ; 300  $\mu\text{m}$ - $\Delta$  = 36.3  $\mu\text{m}$ ).

Heat liberated during the heterogeneous reaction is expended for the most part on heating of the entire mass of the particle of metal by thermal conductivity and is partly lost to the surrounding environment. Stresses arise in the oxide film due to the increase in the temperature of the heater (in the experiments heating was dynamic) and as a result of the heterogeneous reaction, taking place on the surface of the metal particles. The stresses increase during diffusion of oxygen into the particles and as a result the oxide film cracks and is destroyed. Nitrogen is present in the air which also contributes to and accelerates the destruction of the oxide film [2,3]. These phenomena lead to the intensive growth of the thickness of the oxide film on particles of zirconium and titanium (up to a temperature of 900°). After cooling the zirconium particles are a dull white color and the titanium particles a dull yellow.

Along with the metals a study was made of the pre-ignition process of zirconium hydride. From published data it is known that zirconium hydride ignites easily [4], but the ignition temperature of the hydride is higher than that of pure zirconium [5].

Under conditions of the experiments the behavior of zirconium hydride particles resembles the behavior of zirconium and titanium particles but with a more complex mechanism; with an increase in the temperature from 20 to 200° an insignificant growth of the diameter of the particles is noted (100  $\mu\text{m}$ -  $\Delta$ =0.2  $\mu\text{m}$ ; 200  $\mu\text{m}$ -  $\Delta$ =0.5  $\mu\text{m}$ ; 520  $\mu\text{m}$ - $\Delta$  =3  $\mu\text{m}$ ). In the temperature interval from 200-300° lies the beginning of the heterogeneous reaction. Further heating to 900° leads to an increase in the size of the particles (100  $\mu\text{m}$ -  $\Delta$ =7.5  $\mu\text{m}$ ; 200  $\mu\text{m}$ -  $\Delta$ =18.2  $\mu\text{m}$ ; 520  $\mu\text{m}$ -  $\Delta$ =27.5  $\mu\text{m}$ ).

Oxidation probably proceeds according to the following scheme: dehydrogenation from the surface of the particle; interaction of the metal with oxygen. The described mechanism of oxidation

takes place during the entire process of heating. This also explains the relatively small increase in the size of the zirconium hydride particles. This mechanism is also confirmed by the decrease in the diameter of the particles during cooling as a result of the continuing decomposition of the hydride. After cooling the zirconium hydride particles have the color of zirconium oxide.

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