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CORROSIVE INFLUENCE OF DIPHENYL ON METALS AND
OXIDES

Yu. F. Bychkov, et al

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By

Yu. F. Bychkov, I. D. Laptev, and A. N. Rozanov

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CORROSIVE INFLUENCE OF DIPHENYL ON METALS AND OXIDES

By: Yu. F. Bychkov, I. D. Laptev, and
A. N. Rozanov

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ABSTRACT: The authors investigated the transformations which can occur with reactor materials exposed to biphenyl and evaluated the effect of such factors as the presence of admixtures and the degree of pyrolytic decomposition. The hydride mechanism of corrosion was investigated first with respect to uranium and its alloys with molybdenum. The corrosion tests with biphenyl were carried out in containers made of 1Kh18N9T stainless steel. After evacuating the containers with the specimens, they were pressurized in an arc furnace in an atmosphere of pure argon. The changes undergone by a variety of metals, alloys and oxides are tabulated. The authors also investigated the effect of admixtures of water (0.0004-0.7 wt.%) and sulfur (1-3 wt.%) to biphenyl on its corrosive properties. The results indicate that metals and oxides are subject to the following types of transformations in biphenyl: reduction of oxides and hydroxides to the metal or another oxide; formation of hydrides; formation of carbides; and oxidation. Admixture of water to biphenyl in amounts greater than 0.2% was shown to hasten the corrosive failure of aluminum A-1 and SAP-1 alloy and lead to intercrystalline corrosion, as well as embrittling and lowering the strength of the metals. Corrosion was also accelerated by sulfur. Admixtures of water do not affect SAP-1 alloy as much as aluminum AD-1. Original article has: 7 figures and 1 table. English translation: 16 pages.

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Block	Italic	Transliteration	Block	Italic	Transliteration
А	<i>А</i>	A, a	Р	<i>Р</i>	R, r
Б	<i>Б</i>	B, b	С	<i>С</i>	S, s
В	<i>В</i>	V, v	Т	<i>Т</i>	T, t
Г	<i>Г</i>	G, g	У	<i>У</i>	U, u
Д	<i>Д</i>	D, d	Ф	<i>Ф</i>	F, f
Е	<i>Е</i>	Ye, ye; E, e*	Х	<i>Х</i>	Kh, kh
Ж	<i>Ж</i>	Zh, zh	Ц	<i>Ц</i>	Ts, ts
З	<i>З</i>	Z, z	Ч	<i>Ч</i>	Ch, ch
И	<i>И</i>	I, i	Ш	<i>Ш</i>	Sh, sh
Й	<i>Й</i>	Y, y	Щ	<i>Щ</i>	Shch, shch
К	<i>К</i>	K, k	Ъ	<i>Ъ</i>	"
Л	<i>Л</i>	L, l	Ы	<i>Ы</i>	Y, y
М	<i>М</i>	M, m	Ь	<i>Ь</i>	'
Н	<i>Н</i>	N, n	Э	<i>Э</i>	E, e
О	<i>О</i>	O, o	Ю	<i>Ю</i>	Yu, yu
П	<i>П</i>	P, p	Я	<i>Я</i>	Ya, ya

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

CORROSIVE INFLUENCE OF DIPHENYL ON METALS AND OXIDES

Yu. F. Bychkov, I. D. Laptev, and A. N. Rozanov

Introduction

Organic heat-transfer agents, of interest for atomic power engineering, are, as a rule, compounds having complex structures or their mixtures, whose composition becomes still more complicated in the course of pyrolysis and radiolysis. In connection with this, the influence of organic compounds with comparatively simple structures, for instance diphenyl, on metals is of interest for the understanding of the processes of corrosion in liquids of a more complicated structure.

The goal of the investigations conducted by the authors consisted of clarification of transformations which can occur with materials in diphenyl and appraisal of the influence of such factors as the presence of impurities and the degree of pyrolytic decomposition. The hydride mechanism of corrosion, one of the most important in organic heat-transfer agents, was studied with uranium and its molybdenum alloys.

Corrosion tests in diphenyl were conducted with containers made

of stainless steel of brand [1Kh18N9T] (1X1849T). Into the container we poured 70-90 cm³ of diphenyl. After evacuation the specimens were hermetically sealed in an arc furnace in an atmosphere of pure argon. Then for carrying out the tests we placed the containers in furnaces, whose temperatures were maintained in the assigned interval.

Reducing Power of Diphenyl

For studying the processes occurring with materials in diphenyl, we determined the change in structure of powders of different metals and certain compounds, especially oxides, after the prolonged influence of diphenyl at high temperatures. (The use of powders of the investigated materials as specimens in the experiments was occasioned by the desire to quickly obtain the necessary results. The well-developed surface of powder specimens promoted this.) As is known, during the decomposition of diphenyl atomic hydrogen and hydrocarbons will be formed, which at the moment of separation can interact with materials more actively.

The degree of decomposition and the quantity of gases separated during decomposition depend strongly on the test temperature. Tests on the powders were conducted in the wide interval of temperatures from 320° to 450°C; the duration of holding was 240 h at 450°C and ~500 h at 370° and 410°C. After prolonged holding at the temperatures 320° and 370°C the properties of the diphenyl barely changed and the quantity of high-boiling residues (boiling at a temperature higher than the boiling point of diphenyl, 255°C) was very small (Fig. 1). After holding of the diphenyl at 420°C there was formed a large quantity of highly-volatile gaseous and liquid products which ignited at lower temperatures (~550°C) than the initial diphenyl (650°C).

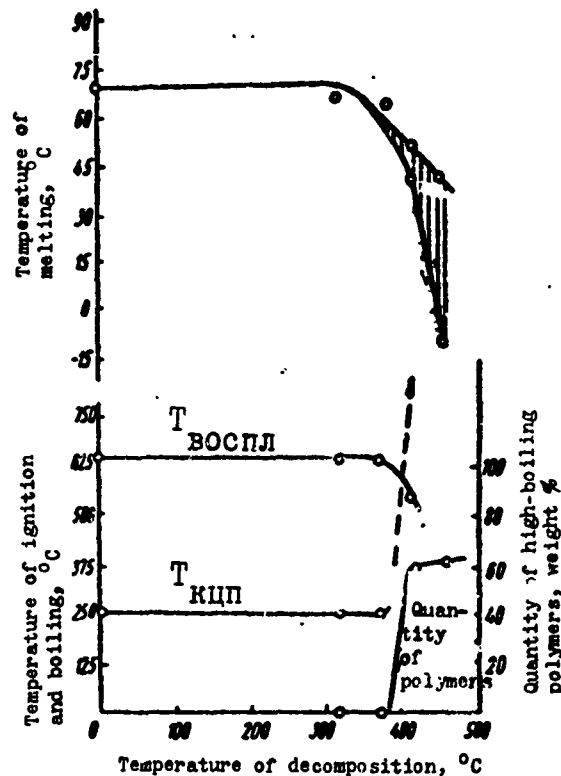


Fig. 1. Influence of holding of diphenyl at 320°-410°C (500 h) and at 450°C (240 h) on the melting, ignition, and boiling points and also on the quantity of high-boiling polymers formed (weight %).

The content of high-boiling polymers in diphenyl held at 420°C was around 60%. The diphenyl was subjected to still greater changes at a temperature of 450°C. After decomposition it was a mixture of liquid and solid phases, which melted in the wide interval of temperatures from -10 to 40°C.

With help of X-ray diffraction analysis of the structure of the metal powders and of the compounds after holding in diphenyl some peculiarities of corrosion were revealed. We investigated the change in structure in [the following] powders: the metals Nb, Zr, Ti, Ta, Fe, and Al; alloy [SAP-1] (САП-1); powders of [APS-1] (АПС-1),

from which alloy SAP-1 is prepared; oxides UO_2 , U_3O_8 , Ta_2O_5 , Nb_2O_5 , ZrO_2 , WO_3 , MoO_3 , Al_2O_3 , and TiO_2 ; uranium carbide; zirconium and hydride. The oxides were prepared by the oxidation of the metals in air at various temperatures, depending upon the heat resistance of the metal and the volatility of the oxides, and then the oxides were crushed in a mortar. Every powder was placed in a separate bore of a special insert made from alloy SAP-1 in which we drilled 16 pockets 15 mm in depth and 3 mm in diameter.

The results of the investigations are given in the table.

Reduction was observed in the comparatively unstable oxides: powdered APS-1 containing aluminum hydroxide was turned into pure aluminum. The mixed oxide of uranium U_3O_8 was reduced to UO_2 . The tungsten oxide was also subjected to changes; the products of its transformation were not identified, but they were neither pure tungsten nor tungsten carbide. The more durable oxides Al_2O_3 , ZrO_2 , TiO_2 , Ta_2O_5 , Nb_2O_5 , and UO_2 were stable in diphenyl which was strongly decomposed at 410°C and the oxides Nb_2O_5 , Ta_2O_5 , TiO_2 , ZrO_2 , and Cr_2O_3 were not changed even after holding at 450°C .

Thus at a temperature of 450°C in diphenyl reduction occurs of oxides whose free energy of formation at this temperature is less than approximately 80-90 kcal/g-atom oxygen. Such oxides include oxides of the metals Co, Ni, Fe, Mo, V, and W; tantalum oxides; and (close to the latter in stability) oxides of Mn and Cr.

A number of metals form hydrides in diphenyl: niobium is transformed at 370°C (500 h) into NbH with a lattice parameter of 3.42 \AA . Tantalum is turned into TaH with a lattice parameter of 3.42 \AA ; however, on the X-ray photograph of tantalum held in diphenyl

Table of the Change of Structure of Powders of Metals and Other Compounds after Holding in Diphenyl at 370°-450°C.

Initial product	Conditions of test	Final product	Initial product	Conditions of test	Final product
No change in structure			Reduction		
UO ₂	450°, 240 h 410°, 540 h 370°, 500 h	UO ₂	U ₃ O ₈	450°, 240 h 410°, 540 h 370°, 500 h	UO ₂
ZrO ₂	450°, 240 h 410°, 540 h 370°, 500 h	ZrO ₂	АПС-1	450°, 240 h 410°, 540 h 370°, 500 h	Al
TiO ₂	450°, 240 h 410°, 540 h 370°, 500 h	TiO ₂	Carbide formation		
Ta ₂ O ₅	450°, 240 h 410°, 540 h 370°, 500 h	Ta ₂ O ₅	MoO ₃	410°, 540 h	MoC+MoC ₂
Nb ₂ O ₅	450°, 240 h 410°, 540 h 370°, 500 h	Nb ₂ O ₅	Ti	450°, 240 h 410°, 540 h 370°, 500 h	TiC Ti+TiC Ti+TiC ₂
Cr ₂ O ₃	450°, 240 h 410°, 540 h 370°, 500 h	Cr ₂ O ₃	U	420°, 200 h	UC+UH ₂
			Hydride formation		
Fe	450°, 240 h 410°, 540 h 370°, 500 h	Fe	Zr	450°, 240 h 410°, 540 h 370°, 500 h	ZrH
UC	450°, 240 h 410°, 540 h 370°, 500 h	UC	Nb	450°, 240 h 410°, 540 h 370°, 500 h	NbH
ZrH	450°, 240 h 410°, 540 h 370°, 500 h	ZrH	Ta	450°, 240 h 410°, 540 h 370°, 500 h	TaH
Al	410°, 540 h	Al	U	370°, 500 h	UH ₃

we also observed three unidentified lines. Zirconium agreeing with many data also forms a hydride.

For the clarification of the possibility of zirconium hydride in the decomposed diphenyl being transformed into a carbide of zirconium, we investigated the powder of zirconium hydride obtained during the 2-hour holding of a strip of Zr at 900°C in an atmosphere of hydrogen. After holding in diphenyl at 450°C this zirconium hydride slightly changed its structure: on the X-ray photograph a few new lines appeared. Powder of unalloyed zirconium held in diphenyl at 450°C had precisely the same structure as did the zirconium hydride held in diphenyl, i.e., in both cases the same stable product was formed in the decomposed diphenyl. The most intense lines of the corrosion product coincide with the lines of the initial zirconium hydride. Besides this phase, the product contained other unknown compounds but not zirconium carbide (possibly carbohydrides).

In diphenyl, as it turned out, the formation of the carbides of several metals also occurred. The powder of titanium held at 410°C was partially turned into titanium carbide; on the X-ray photograph of this powder were seen both the lines of titanium carbide and the lines of titanium, whose intensity was higher. Possibly hydrogen was dissolved in the titanium and the powder in this case consisted of a mixture of titanium carbide and titanium α -hydride (or of a mixture of TiC + Ti). After holding in diphenyl at 450°C titanium was completely changed into titanium carbide.

The formation of carbides was observed not only during holding of titanium in diphenyl, but also during holding of uranium. A strip of pure uranium was turned into powder of the hydride UH₃

after holding at 370°C and into the uranium monocarbide UC at 420°C . The possibility of further interaction of the uranium monocarbide with the products of the pyrolysis of diphenyl and of the formation of other uranium carbides was checked by holding UC powder in diphenyl.

The UC powders were obtained by grinding an ingot of uranium monocarbide (which had been smelted in an arc furnace) in a mortar. During the grinding some oxidation of the uranium monocarbide occurred, which led to the appearance of several lines of uranium dioxide on the X-ray photograph of the powder obtained. Holding this powder in diphenyl at 450°C did not lead to a change in structure: the powder contained approximately the same quantity of UC and UO_2 as did the initial; no new lines were observed after holding. Thus, even under conditions of strong pyrolysis of diphenyl uranium monocarbide is a stable phase.

In diphenyl, just as in other organic media, oxidation can occur when it contains impurities of water or oxygen. The authors established that after 520 h of holding at 410°C in diphenyl with an addition of 0.2% water even a fairly stable alloy of uranium with 9% Mo was turned into UO_2 . It was also revealed that on the surface of magnesium in diphenyl at 370°C and 410°C a friable film containing MgO will be formed. Other authors [1,2] observed the formation of oxidized films ZrO_2 and Fe_3O_4 on zirconium and stainless steel and of MgO film on Al - Mg alloys.

The data obtained to the fact that different processes of transformation of metals and compounds occur in diphenyl: reduction, hydride formation, carbide formation and oxidation.

Hydride Corrosion of Uranium and Its Alloys with Molybdenum

We studied the kinetics of the corrosion destruction of uranium in diphenyl at 320°C (Fig. 2) and for alloys with Mo at 420° and 450°C.

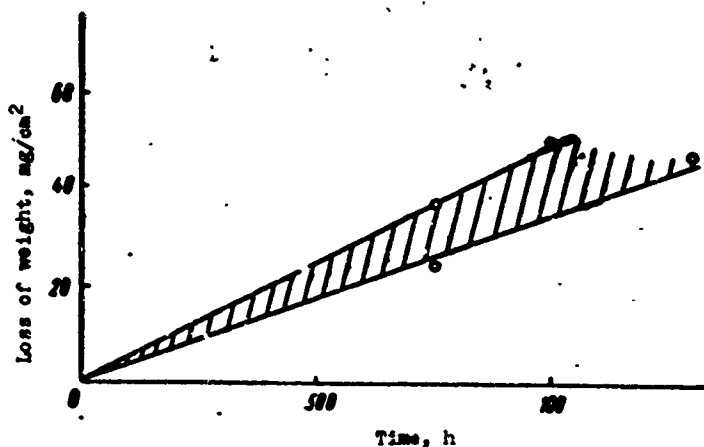


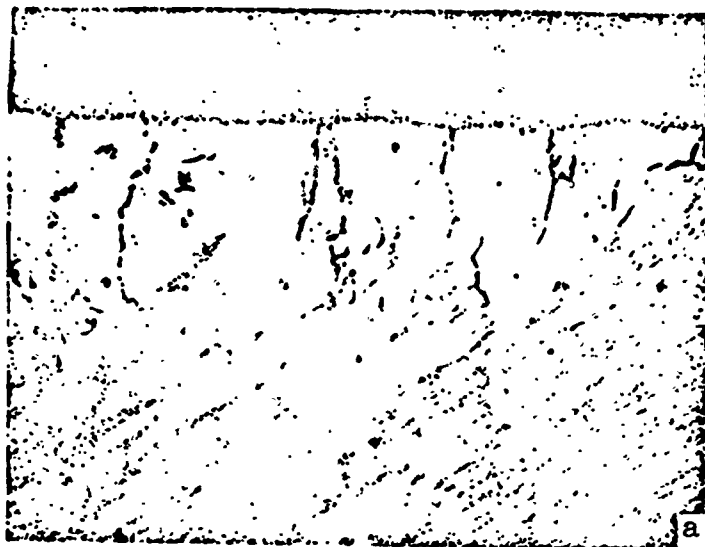
Fig. 2. Weight loss of uranium after holding at 320°C in diphenyl.

uranium were subjected to etching in 10 vol. % H_2SO_4 for 30 minutes at 50°C for removal of the products of corrosion. The metal was not dissolved. The specimens of uranium and of the alloys after holding at 420° and 450°C were not subjected to etching for removal of corrosion products.

At 320°C the unalloyed uranium is destroyed at an approximately constant rate at the expense of the formation of the hydride. With an increase of the temperature to 420°C the speed of the destruction of uranium noses sharply, due to the increase of the degree of decomposition of the diphenyl and the change of the corrosion mechanism (formation of the monocarbide and not the hydride).

Uranium in diphenyl is subjected to intercrystalline destruction (Fig. 3); the depth of penetration of the hydride increases with an increase in temperature and time of holding. After 500 h at 370°C the depth of destruction is 150 μ ; 420°C after 50 h the depth of

We conducted the investigation on strips 0.5 mm thick of uranium and its alloys with 3.5; 6, and 9% Mo, obtained by hot rolling in vacuum from ingots smelted in an arc furnace. After holding in diphenyl at 320°C, the specimens of



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Fig. 3. Microstructure of unalloyed uranium after holding in diphenyl: a) at 370°C for 500 h; b) at 420°C for 200 h (the slides are not etched, $\times 270$).

corrosion 100 μ ; after 200 h the uranium hydride penetrates to a depth of 400 μ . The microhardness of the grains on the surface of the uranium specimen which was held at 420°C for 50 h and at 370°C for 500 h was the same as that in the middle of the specimen or even somewhat lower (20 kg/mm²), which attests to the absence of the formation of a solid solution of hydrogen in the uranium.

After holding in diphenyl at 420°C for 200 h the tensile strength of the uranium was 28 kg/mm^2 , i.e., it was considerably lower than for the specimens held at those same regimes in argon (94 kg/mm^2). The prolonged holding of uranium for 400 h at 450°C in diphenyl led to a transformation of laminar specimens into powder.

The formation of the uranium monocarbide at 420° and 450°C apparently occurs by means of the interaction of the products of pyrolysis with the earlier formed uranium hydride and not by means of interaction directly with uranium, inasmuch as the reaction product of uranium and diphenyl is a powder. The process proceeds in an analogous manner in other organic compounds.

According to the investigations of Troutner [3] the process of the formation of UC in another liquid - monoisopropyldiphenyl - is autocatalytic, since during the formation of the uranium monocarbide hydrogen is separated; it can interact with the uranium, transforming it into uranium hydride. Transformation of the uranium in the organics proceeds by the following chain of reaction:

1. Organic compound \rightarrow changed organic compound $+ \text{H}_2$.
2. $\text{U} + 3/2\text{H}_2 \rightarrow \text{UH}_3$.
3. $\text{UH}_3 + \text{organic} \rightarrow \text{UC} + 3/2\text{H}_2 + x\text{H}_2$.

Alloys of uranium with molybdenum were tested under stricter conditions than the unalloyed uranium: at 420°C for 240 h and at 450°C for 160 h (Fig. 4). As is known [4], after hot rolling and fast cooling the uranium alloy with 3.5% molybdenum has an α -structure; the alloy with 6 wt.% after hardening has a γ -structure; the alloy with 9 wt. % of molybdenum has the structure of a highly stable γ -phase. The corrosion influence is strongly noticeable

during great degrees of diphenyl decomposition on the alloy with 3.5% of molybdenum. For the remaining alloys the change of weight was the same as in argon. This testifies to the high corrosion stability of these alloys in diphenyl.

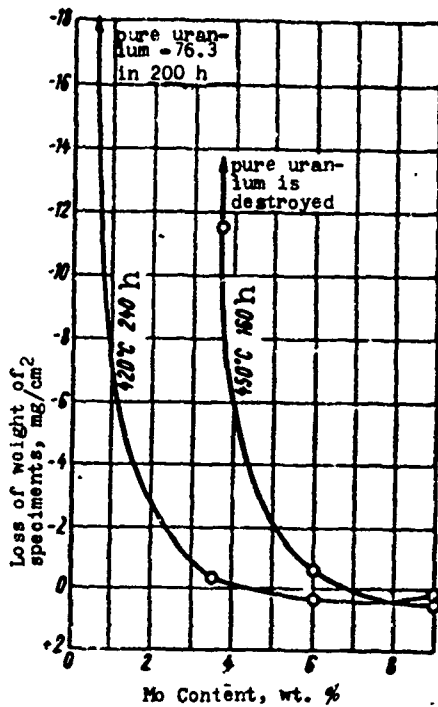


Fig. 4. Influence of alloying uranium with molybdenum on weight loss after holding in diphenyl at 420°C for 240 h and at 450°C for 160 h.

The corrosion of the uranium alloy with 3.5% of molybdenum has a strongly pronounced intercrystalline character: the metal near the boundaries of the grains is corroded and in these places there occurs the formation of a new phase. In the remaining alloys no changes of structure were revealed. The microhardness of the alloys after corrosion was the same on the surface of the specimen as in the deep layers. The decrease in tensile strength to 40 kg/mm² of the uranium alloy with 3.5% molybdenum after holding for 160 h at 450°C confirms the presence of intercrystalline corrosion.

The surface of a rolled specimen made from the uranium alloy with 9 wt. % molybdenum after holding in diphenyl for 400 h at 450°C remained absolutely pure; no traces of destruction whatsoever were revealed by visual inspection and the increase of weight totalled only 0.2 mg/cm². From this it follows that the uranium alloy with 9 wt. % molybdenum possesses very good stability in diphenyl even when holding for 400 h at 450°C.

The corrosion influence of different fractions formed by pyrolysis of diphenyl can be unequal, because different fractions contain diverse quantities of highly volatile products and impurities. We also investigated the corrosion of uranium specimens in a liquid fraction which was obtained by filtration of diphenyl subjected to pyrolytic decomposition at 420°C for 500 h. The weight loss after holding for 160 h at 420°C in this liquid was 4.6 mg/cm², i.e., it had almost the same value as the weight loss after corrosion in diphenyl under the same conditions. After holding in the liquid products of the pyrolysis of diphenyl, we observed intercrystalline destruction of the uranium specimen to a depth of 250 μ. In one experiment, the samples (located in a gas phase above the diphenyl at 370°C for 500 h and at 420°C for 300 h) had the same weight loss as the specimens held in diphenyl; in another experiment the lower part of the uranium sample located in the diphenyl underwent somewhat greater corrosion destruction than the upper, which was in the gas phase. This can be explained by the fact that the atomic hydrogen separated during pyrolysis of the diphenyl interacted more actively with the uranium located in the diphenyl and the molecular hydrogen in the gas phase was less active.

Proceeding from this, it is possible to consider that the corrosion in the gas phase above the diphenyl occurs slower than the corrosion in the actual liquid, but at great degrees of decomposition of the organic and with prolonged holding identical results are obtained.

Influence of Impurities of Water and Sulfur
in Diphenyl on Corrosion

In technical lots of organic heat-transfer agents, as is known, there is a certain quantity of water. Inasmuch as in the process of the work of a reactor trickle feeding of fresh coolants occurs. The impurities of water can influence the destruction of the shell of fuel elements.

The authors investigated the influence of additions of water to diphenyl (in the quantities 0.0004, 0.01, 0.04, 0.2, 0.5, and 0.7 wt. %) on the stability of aluminum and the alloy SAP-1, which in the absence of these additives possesses high stability in diphenyl. Duration of holding of plates of these metals was 500 h. Additions of water higher than 0.1% had a strong influence on the speed of destruction of aluminum and alloy SAP-1. After holding the outer appearance of the specimens changed greatly. On the specimens of aluminum we observed a change in color of the corrosion film with an increase in the quantity of water (from silvery-white to gray) and the formation of bubbles, whose quantity increased with an increase in water content. The quantity of bubbles on alloy SAP-1 also increases with an increase in water content. They were disposed along the direction of rolling, in some places the bubbles were detached from the surface. With an increase in water content in the diphenyl the addition of weight of aluminum and alloy SAP-1 rises sharply (although a part of the products of corrosion crumbles), and therefore magnitudes obtained for the weight increase were lower than those which in actually occurred. After holding there occurs a decrease in tensile strength and embrittlement of the plates (Figs. 5 and 6). It is characteristic that additions of water have

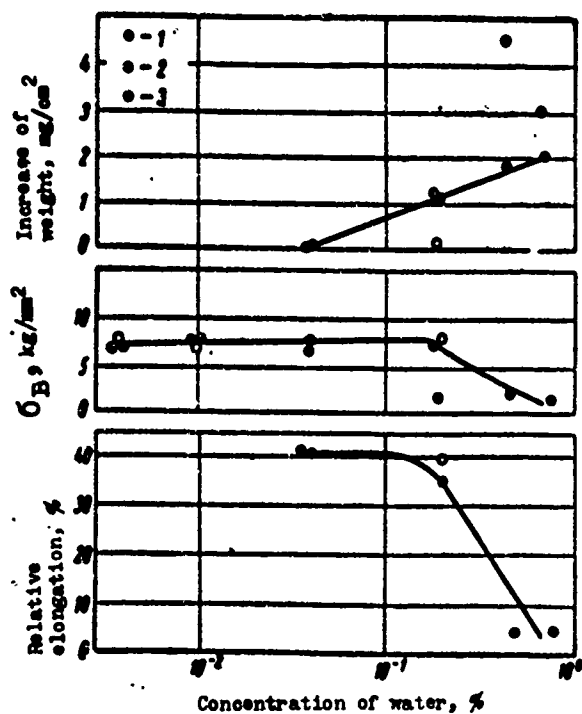


Fig. 5. Influence of additions of water to diphenyl on properties of aluminum AD-1. Conditions of holding: 1 - 410°C, 500 h; 2 - 370°C, 500 h; 3 - 320°C, 500 h.

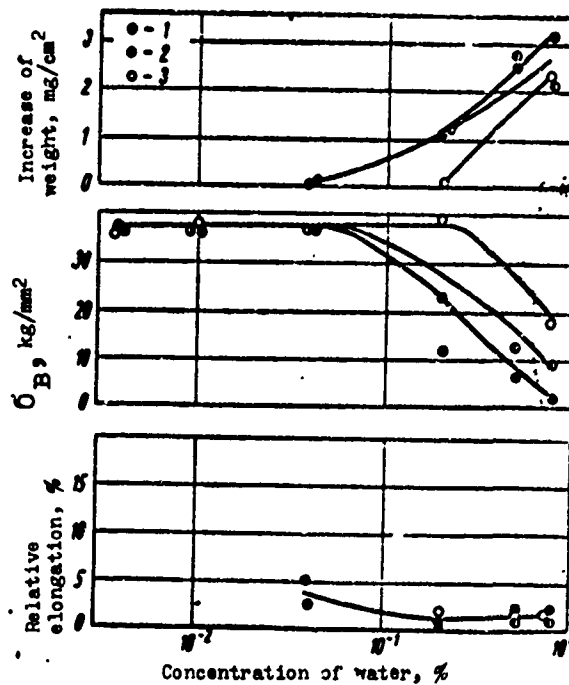
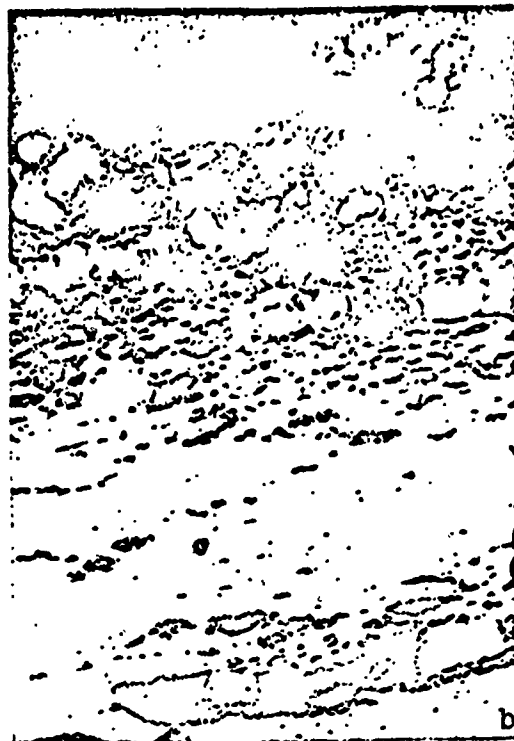


Fig. 6. Influence of additions of water to diphenyl on properties of alloy SAP-1. Conditions of holding: 1 - 410°C, 500 h; 2 - 370°C, 500 h; 3 - 320°C, 500 h.

less effect on the alloy SAP-1 than on aluminum: the depth of the intercrystalline destruction of alloy SAP-1 is 0.3 mm; a plate of aluminum 1 mm thick is corroded right through after holding at 410°C (Fig. 7). These results agree with work [1], in which it was shown that alloys of the SAP type are more stable than aluminum in a medium of organic matter with an impurity of water. Thus, alloy SAP-1 possesses a higher corrosion stability than aluminum in diphenyl with additions of water.

We also studied the influence of sulfur on corrosion of aluminum and alloy SAP-1 in diphenyl. Plates of the metals were held in containers having around 80-90 g of diphenyl with additions of sulfur in quantities of 1 and 3 wt. %. In order to exclude the



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Fig. 7. Microstructure of aluminum AD-1 (a) and alloy SAP-1 (b) after holding in diphenyl with addition of 0.5 wt. % water at 410°C for 500 h (slides are not etched, $\times 340$).

direct interaction of sulfur with the specimens, the latter were placed on a special support 5-7 mm high and the melted sulfur in the case of immiscibility should be found on the bottom of the container. In every container we placed one plate each with dimensions of 60 \times 35 mm.

After holding at 450°C for 540 h the aluminum specimens lost 0.02 mg/cm² in weight with 1% sulfur in the diphenyl and 0.01 mg/cm² with 3% sulfur; the plates of alloy SAP-1 under the same conditions changed their weight respectively to 0.01 mg/cm² and +0.01 mg/cm², i.e., the changes in weight were small.

Conclusion

I. Metals and oxides in diphenyl are subjected to the following forms of transformations:

- 1) reduction of the oxides and hydroxides to the metal or another oxide;
- 2) the formation of hydrides;
- 3) the formation of carbides;
- 4) oxidation.

II. Additions of water to diphenyl in quantities higher than 0.2% accelerate the corrosion destruction of aluminum [AD-1] (АД-1) and alloy SAP-1 and lead to intercrystalline corrosion; they also embrittle the metals and lower their strength. Impurities of water influence alloy SAP-1 to a lesser degree than they do aluminum AD-1.

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