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UREA HIGH TEMPERATURE GREASE FOR AIRCRAFT

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Abstract

Purpose: *The research results of the synthesis of multipurpose urea grease are based on synthetic oils for use in a wide temperature range, suitable for use in aviation engineering.* ***Methods:*** *The interaction of polyisocyanate with octadecylamine and benzylamine in a mixture of polyalphaolefin oil with pentaerythritol esters and fatty acids of the C_5-C_9 fraction was synthesized polyurea – thickener of urea greases. Its structure was established by IR spectroscopy. The method of derivational analysis was used to determine the upper temperature limit of grease application. The properties of grease are investigated by standard methods.* ***Results:*** *IR spectra of urea grease indicate that was applied for the synthesis of the thickener the ratio of reagents, leads to their full consumption during the reaction and allows to obtain urea with the maximum output. The nature of the curves of differential thermal analysis and thermogravimetry indicates that the upper temperature limit for the application of urea grease made on a mixture of synthetic oils is at 200 °C. It is established that the synthesis temperature and the ratio of the components of the thickener determine the thermal, rheological and mechanical properties of the system. By varying the molar ratio of the components of the urea thickener, it was possible to create a mechanical and colloidal stable lubricanting composition. The grease does not freeze to minus 50 °C, which is confirmed by the low viscosity at this temperature and the value the starting and the running torque of rotation of bearing. Applications of a multifunctional additive package allowed to enter a new qualitative level of tribological characteristics and resistance to oxidative conversions.* ***Discussion:*** *Researches shown that the properties of urea grease depend on not only on the nature and the ratio of the components of the urea thickener and the parameters of the process of its synthesis, but also on the balance of the lubricating composition as a whole with properly selected base oils and an additive package. The developed product with a high level of tribological, antioxidant and ecological characteristics meets the modern requirements for aviation greases and can be recommended for using in harsh operating conditions over a wide temperature range.*

Keywords: aviation grease; isocyanate; amine; urea thickener; thermooxidative stability; tribological characteristics

1. Introduction

In metallurgy, mining or silica industries at high temperature in the friction units is necessary to apply an increased amount of grease and replace it more frequently. Such methods cannot be used in military technique and aviation, where temperature conditions are especially harsh and defect of friction unit can lead to disaster. In airplanes the high temperature occurs in jet engines and high-speed bearings. Low temperatures occur at high altitudes and in Arctic areas. The need for highly efficient multi-functional greases is increased in a result of recent achievements in the field of aircraft construction. The speed of the aircrafts increases, increases thermal performance for certain parts. The friction units of aircrafts with speeds of over 2 M can be heated to 200-250 °C [1]. As a result, toughening mode of operation of grease in the bearings ailerons, stabilizers and rudders. Their drivers also work in very stressful conditions. Most aircraft units are not exposed to twisting movement with high speeds, but feel strong vibrational loading. Aircraft greases should be able to work throughout the temperature range - on land and in the air. Greases which are based on oils may be used only under normal temperatures. In the case of expanding the range of operating temperature, e.g., from minus 50 to plus 170 °C, greases on this basis are unsuitable. Many thickeners, which successfully operate at 120-150 °C, cannot withstand higher temperatures, and structure of greases collapses. Another problem is the matching of stable and effective, in the range of operating temperatures, antioxidant and anti-wear additives. Compatible of such additives with synthetic dispersion medium is explored extremely insufficient.

Modern aviation greases are high-temperature products with a drop temperature above 250 °C and losses on evaporation at 200 °C, which not exceeding 5%, are resistant to water, non-aggressive corrosive, mechanically stable, mostly 1-2 grade on a scale penetrations NLGI.

2. Analysis of Research and purpose

Finding and developing new types of thickeners are usually caused by rising requirements for greases. Synthesis and patenting in 1955 by the American company Standard of Indiana arylsubstituted urea as a thickener of greass was due to lack of those times is chemically and thermally stable thickeners for silicone oils. Soap, mainly calcium, greases and greases on inorganic thickeners do not provide the necessary resource of friction units of aviation technology in a wide range of temperatures, pressures and speeds. Poliurea or urea greases (UG) were non-corrosive, high-fusible, chemical- and heat-sable. Since then and until today there was the interest of developers in this type of plastic thixotropic colloidal systems. Researchers from around the world for the synthesis of di-, three-, tetra- and poliurea thickener used different combinations of amines and isocyanates, represented different mechanisms of their synthesis and induced different properties of the final lubricating compositions [2-5]. The most simple but effective thickener, which Japanese manufacturers UG give preference [6] considered diurea - product isocyanate interaction with one or two secondary amines. Greases, which are made on diurea, are characterized by a wide temperature working range and the required level of tribological and rheological characteristics, stable at high temperatures, able to work in the presence of moisture.

In view of the above, the purpose of work was developing new backfill multipurpose grease on synthetic oils, which are thickened by diurea for a wide temperature range of aviation engineering.

3. Experimental

As the dispersion medium of UG was used mixture of polyalphaolefin oil (PAO) with pentaerythritol esters and fractions of fatty acids C_5-C_9 (EP). Instead of toxic monomeric isocyanates, which require special safety measures, we used the so-called "grey" diphenylmethane diisocyanate (Crude MDI) or polyisocyanate (PIC), with a mass fraction of isocyanate groups of 35–38 %, to obtain a UG urea thickener. The interaction PIC with octadecylamine and benzylamine was synthesized product containing group -NH-CO-NH-, which positively influence on the UG lubricating properties and performance of the whole system [7].

UG is synthesized by the known technology [8]. It’s prepared individual solutions of PIC and mixtures of amines in the environment of PAO. Heated to 70 °C and mixed in a reactor, which is equipped with heating and mixing device. To the formed suspension, the estimated number of PE added and heated to a certain temperature in the range of 100-200 °C with holding 30 min. The samples were cooled to 20 °C and homogenized in a laboratory three-roll mill.

IR-spectrum of synthesized compounds in the range of 4000-400 cm^{-1} was recorded using IR-spectrometer with Fourier transform Thermo Nicolet Nexus FT-IR (USA). To determine the temperature of the upper boundaries of UG was used derivatographic the method of analysis, which are based on the simultaneous measurement of mass and enthalpy of material during its heating. The study was conducted in an atmosphere of static air through the unit Q-1500D (Hungary) with computer registration of data. Derivatography allows determines the temperature, at which occur physical and chemical transformations in the thickener. During derivatographic analysis of the sample of grease simultaneously record curves, differential thermal analysis (DTA), thermogravimetry (TG) and differential thermogravimetry (DTG).

To enhance level of the thermo-oxidative stability to UG composition introduced application Borine – are processed by boric acid the product condensation 2,6-di-tert-butylphenol and alkylphenols with formaldehyde and ammonia. To improve the tribological characteristics of UG is used anti-wear package and anti-seize additives consisting of dialkyldithiophosphate of zinc and a mixture of sulfide olefins.

Temperature indicators of dropping and colloidal stability of UG determined by ASTM D566, ISO 2176 and GOST 7142. Mechanical stability (MS) of UG evaluated by the change in penetration rate after extended mixing (P_2 - P_1 = ΔP). According to ASTM D217, ISO 2137 standard greases break down in the mixer of penetrometer. P_1 determined after 60 and P_2 - after 100 000 double strokes. Changing the status of the consistency of grease during long mixing characterizes the susceptibility or resistance of grease to thixotropic destruction. The stability of grease samples to oxidation analyzed for acid number changing after prolonged heat treatment according to GOST 5734, and their tribological properties - critical load (P_c) and welding load (P_w) was determined on four-ball friction machine to GOST 9490.

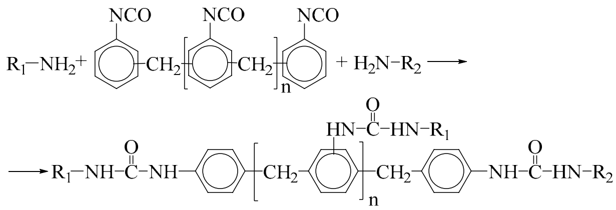
To determine the functionality of grease at high temperatures conducted its test on the stand SETA-1860, corresponding method FTMS 719B-331.1 US federal standards. This high speed bearing stand, consisting of three identical units that include thermostatic chamber with heaters, bearing assembly and motor with belt drive to spindle. Criteria for evaluating performance of grease are regular term of the bearing filled it to its failure or the appearance of one of the following factors: the bearing temperature rise above the set standards, and its stiffness of oil and its leakage from bearing, increased noise of bearing. UG investigated in bearing 70-204K3 at a radial and axial loads of 23 N, the rotational speed of 10,000 min^{-1} and a temperature of 150 °C.

To study the low-temperature properties of UG was used stand corresponding method ASTM D1478. The stand allows to evaluate starting characteristics bearings with grease at negative temperatures. It identifies two indicators: the starting torque (M_s) - the maximum torque measured at the start of rotation of bearing, and the running torque (M_r) - the 15-s average value of the torque after rotation for a specified period of time (60 min).

Biodegradability of grease evaluated by DSTU 4247:2003 "Petroleum products. The method of determining of biodegradability (CECL 33-A-93, NEQ)".

4. Results and discussion

The process of interaction of PIC with amines occurs on the scheme:



The formation of urea under the above scheme shows the IR spectrum UG (Fig. 1). At UG spectrum no absorption bands of primary amines between 3400-3200 cm^{-1} and the isocyanate groups -N=C=O PIC in the 2280-2230 cm^{-1} [9].

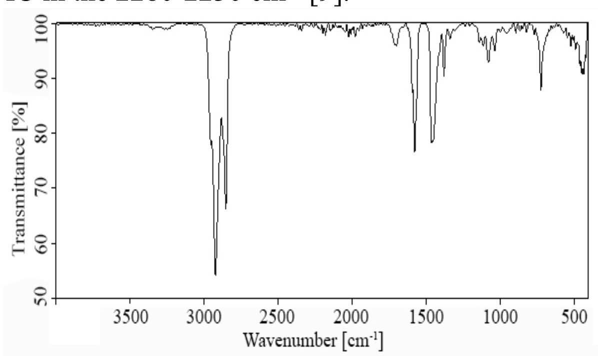


Fig.1. IR spectrum of UG.

At TG curves (Fig. 2) was the initial horizontal section of 200 °C indicates high resistance to lubricant compositions thermal formation. At higher temperatures up to 300 °C is observed sharp loss of grease's weight by intense chemical disintegration and evaporation of its synthetic dispersion medium. On the curves DTA are observed two exothermic effects in the temperature range 300-600 °C, describing the intense thermo-oxidative destruction of dispersion medium and urea thickener. The results make possibility to determine the upper temperature limit UG, which are made by a mixture of synthetic oils at 200 °C.

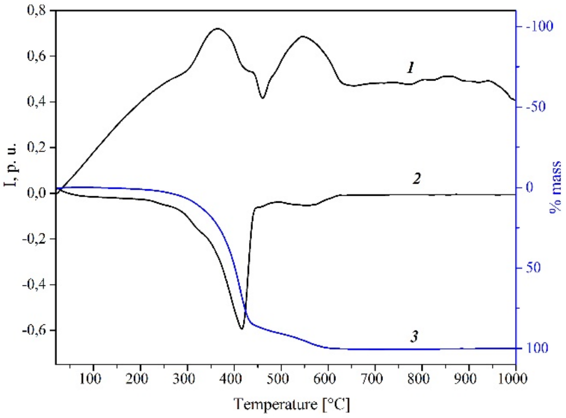


Fig.2. Derivatogram of UG.
1 - DTA curve, 2 – DTG, 3 - TG.

TG curve in the figure indicates 100% loss of UG weight when heated to 600 °C. Diurea does not form ashes, unlike soap greases. This makes profitable benefits to grease on the diurea thickener at application in high temperature conditions. On the friction surfaces are eliminated gome and sludge deposits that extends the life of the equipment.

The researching influence of heat treatment temperature (HTT) on the properties of greasing compositions have found that increase the dropping point temperature of the grease, but deteriorate rheology - colloidal stability, strength, penetration. HTT is optimal in the range of 150-170 °C.

By increasing the proportion of benzylamine in molar ratio of isocyanate : aliphatic amine : aromatic amine significantly improved thermal stability and antioxidant stability of system. An important result of the research was to establish dilution of UG after mechanical destruction, which is characteristic for compositions with high aromatic amines consisting of urea thickener. That is, increasing concentrations of benzylamine leads to poor mechanical stability of UG. Changing the status of UG consistency during prolonged stirring to the value of ΔP = 80-100 units characterized by poor structural stability of the frame to collapse, which could lead to critical dilution of grease and its leakage from the friction unit.

Based on the achieved results, developed an optimal composition of three-component diurea thickener and effective synthesis conditions of thixotropic colloidal urea system with high thermal properties and mechanical stability.

The presence of a significant number of urea -NH-CO-NH- and secondary amine -NH- groups in the molecule of thickener positive effect on the antioxidant properties of UG. In fact, the product of interaction PIC with amines is an inhibitor of group 1, which interrupts the chain oxidation by reaction with peroxide radicals to form polar transition complex $ROO^{\bullet} \bullet\bullet\bullet H \bullet\bullet\bullet In$. To these inhibitors typically include phenol, naphthol, secondary amines, aminophenol, etc. [10]. The inhibitor Borine was highly effective at high temperatures (150-180 °C). Probable the cause is thermal stability of Borine original molecule and its phenoxy radical and products' activity of their transformations in slowing down the process of oxidation [11].

The table shows the results of laboratory and bench tests of developed multipurpose grease.

According to the data of table, UG has a high thermal stability. The dropping point of the grease is 260 °C, evaporation at 200 °C - less than 5%, efficiency in the bearing at high temperatures exceeding 1000 h, that 2 times better than lithium complex greases on the same synthetic basis and with the same additives.

Table

Characteristics of UG

Name of indicator, unit of measure	Test method	Indicator value
Oil separation, %	GOST 7142	8,4
Dropping point, °C	ASTM D566 ISO 2176	260
Evaporation loss, 200 °C, 1 hour, %	GOST 9566	4,5
Increase acid number (150°C, 10 h), mg KOH/g	GOST 5734	0
Viscosity at minus 50 °C, Pa·s	GOST 7163	1490
Torque at minus 50 °C, N·m - M_s - M_r	ASTM D1478	 0,198 0,076
Penetration at 25 °C, $mm \cdot 10^{-1}$ - worked 60 strokes - worked 100 000 strokes - ΔP	ASTM D217 ISO 2137	 300 332 32
Water-washout, 79 °C, %	ASTM D1264	0,5
The functionality on the stand-SETA 1860, hrs	FTMS 719B-331.1	>1060
Tribological characteristics on FBM - P_c, N - P_w, N - D, 392 N, 1 hour, mm	GOST 9490	 1098 2450 0,5
Copper corrosion	GOST 9.080	pass
Biodegradability, %	DSTU 4247	39,8

The using of a multifunctional additive package possible to reach a new level of quality for the tribological characteristics and resistance to oxidative changes. Long-term tests in terms of thermo-oxidative factors (high temperature, copper catalyst and oxygen atmosphere) did not lead to increased acid number. That's why, chain reaction interrupted early emergence of peroxide radicals. Grease also showed relatively high anti-wear properties for the critical load and wear scar diameter (P_c, D) and anti-seizure characteristics by the welding load (P_w).

By varying the molar ratio of components urea thickener, managed to create a mechanically stable lubricating composition of ΔP, which does not more than 40 units. This allows to predict long-term stable operation of UG in friction units without softening and leakage. Designed grease is characterized by high colloidal stability, lack of corrosion activity and water-wash resistance. The grease does not freeze to minus 50 °C, it is confirmed by low viscosity at this temperature and torque value at the starting and a running rotation of the bearing.

New multipurpose grease prevails known commodity analogue on environmental performances, which are defined by DSTU 4247: 2003 "Petroleum products. The method of determining biodegradability (CECL 33-A-93, NEQ)». Indicator of biodegradability compared, for example with grease Maspol (TU U 00149943.489-97) was reduced by almost 10%.

6. Conclusion

Consequently, researches shown that UG properties depend not only on the nature and value components of urea thickener and its synthesis process parameters, but also on the balance lubricant composition as a whole with properly selected base oils and additive package.

Summarizing the results, we can say that designed UG meet the modern requirements of aviation greases and can be recommended in aircraft as multifunctional grease for use in a wide temperature range, under high loads and speeds.

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Високотемпературне уреатне мастило для авіаційної техніки

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Мета: Представлені результати досліджень особливостей синтезу багатоцільового уреатного мастила на синтетичних оливах для широкого температурного діапазону застосування, придатного для використання у авіаційній техніці. **Методи:** Взаємодією поліізоціанату з октадециламіном та бензиламіном у суміші поліальфаолефінової оливи з естерами пентаерітриту і жирних кислот фракції C_5-C_9 синтезовано полісечовину – загусник уреатних мастил. Її будова встановлена методом ІЧ-спектроскопії. Для визначення верхньої температурної межі застосування мастила використано метод дериватографічного аналізу. Властивості мастила досліджені стандартними методами. **Результати:** ІЧ-спектри уреатного мастила засвідчили, що застосоване для синтезу загусника співвідношення реагентів призводить до повного їх витрачання у ході реакції та дозволяє одержати сечовину з максимальним виходом. Характер кривих диференціального термічного аналізу та термогравіметрії вказує, що верхня температурна межа застосування уреатного мастила, виготовленого на суміші синтетичних олив, знаходиться на рівні 200 ^oC. Встановлено, що температура синтезу і співвідношення компонентів загусника визначають термічні, реологічні та механічні властивості системи. Варіюючи мольним співвідношенням компонентів сечовинного загусника, вдалося створити механічно та колоїдно стабільну мастильну композицію. Мастило не застигає до мінус 50 ^oC, що підтверджується низькою в'язкістю за цієї температури та величиною крутних моментів на початку та за сталого обертання підшипника. Застосування багатофункціонального пакету присадок дозволило вийти на новий якісний рівень за трибологічними характеристиками і стійкістю до окиснювальних перетворень. **Обговорення:** Дослідження засвідчили, що властивості уреатного мастила залежать не тільки від природи і співвідношення компонентів сечовинного загусника та параметрів процесу його синтезу, але й від збалансованості мастильної композиції в цілому з правильно підібраними базовими оливами та пакетом присадок. Розроблений продукт з високим рівнем трибологічних, антиокислювальних та екологічних характеристик відповідає сучасним вимогам до авіаційних мастил і може бути рекомендований для застосування в жорстких умовах експлуатації в широкому температурному діапазоні.

Ключові слова: авіаційне мастило; амін; ізоціанат; сечовинний загусник; термоокиснювальна стабільність; трибологічні характеристики

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Высокотемпературная полимочевинная смазка для авиационной техники

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Цель: Представлены результаты исследований особенностей синтеза многоцелевой уреатной смазки на синтетических маслах для широкого температурного диапазона применения, пригодной для использования в авиационной технике. **Методы:** Взаимодействием полиизоцианата с октадециламином и бензиламином в смеси полиальфаолефинового масла с эфирами пентаэритрита и жирных кислот фракции C_5-C_9 синтезировано полимочевину – загуститель уреатных смазок. Ее строение установлено методом ИК-спектроскопии. Для определения верхнего температурного предела применения смазки использован метод дериватографического анализу. Свойства смазки исследованы стандартными методами. **Результаты:** ИК-спектры уреатной смазки свидетельствуют, что примененное для синтеза загустителя соотношение реагентов приводит к полному их расходованию в ходе реакции и позволяет получить мочевину с максимальным выходом. Характер кривых дифференциального термического анализа и термогравиметрии указывает, что верхний температурный предел применения уреатной смазки, изготовленной на смеси синтетических масел, находится на уровне 200 °C. Установлено, что температура синтеза и соотношение компонентов загустителя определяют термические, реологические и механические свойства системы. Варьируя мольным соотношением компонентов мочевинного загустителя, удалось создать механически и коллоидно стабильную смазочную композицию. Смазка не замерзает до минус 50 °C, что подтверджается низкой вязкостью при этой температуре и величиной начального и установившегося крутящих моментов вращения подшипника. Применения многофункционального пакета присадок позволило вийти на новый качественный уровень по трибологическим характеристиками и стойкостью к окислительным преобразованиям. **Обсуждение:** Исследования показали, что свойства уреатной смазки зависят не только от природы и соотношения компонентов мочевинного загустителя и параметров процесса его синтеза, но и от сбалансированности смазочной композиции в целом с правильно подобранными базовыми маслами и пакетом присадок. Разработанный продукт с высоким уровнем трибологических, антиокислительных и экологических характеристик соответствует современным требованиям к авиационным смазкам и может быть рекомендован для применения в жестких условиях эксплуатации в широком температурном диапазоне.

Ключевые слова: авиационная смазка; амин; изоцианат; мочевинный загуститель; термоокислительная стабильность; трибологические характеристики

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