The Removal of Silicone Contaminants from Spacecraft Hardware

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Prepared for

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1. Introduction

Silicones are widely used as elastomeric sealants, adhesives, molding compounds, mold releases, potting compounds, lubricants, and vacuum pumping fluids. Nearly 1200 silicone compounds have been tested for outgassing and listed in the NASA space materials database. The wide-range of uses for silicones is due to their versatile properties. Silicones have high thermal and oxidative stability, their physical properties depend only mildly on temperature, and they are inert with respect to many chemical reactions. Silicones have good dielectric strength and low surface tension as well. For example, silicones make good vacuum pumping fluids because of their high molecular weights, low vapor pressures, and thermal stability. Inertness, stability, and low surface tension make silicones excellent sealants, mold releases, and potting compounds.

Because they are so ubiquitous, silicones are often encountered in manufacturing and ground processing accidents. A typical incident involves the failure of a vacuum valve during thermal vacuum testing of space hardware. This failure causes backstreaming of pumping fluid into the vacuum chamber, contaminating the flight hardware. A recent incident involved the packaging of solar cells in shipping foam that was manufactured using a silicone mold release. Following one year of storage in the foam, the mold release had transferred to, and permeated through, the solar cell structure. This incident resulted in the loss of solar cells for a large number of solar panels. In general, accidental transfer of silicones can occur through dripping, spilling, or spraying onto clean hardware.

The inertness and stability of silicones, properties that give many silicones their desirable characteristics, also make them difficult to remove when they have been deposited inadvertently. Many different approaches have been used to resolve incidents such as those described above. Solvents have been tried, including acetone, methylene chloride, methyl ethyl ketone (MEK), and isopropyl alcohol (IPA). Simple Green[®], citrus cleaners, steam, and vacuum baking have been used. Expensive "designer solvents" such as AK225[®] and Brulin[®] have been applied. These approaches usually achieve some amount of silicone removal, but uncertainties in obtainable cleanliness level have led to major hardware disassembly, followed by meticulous hand wiping, immersion in baths, and extensive rinsing. Painted surfaces have been partially or totally stripped and repainted. In some cases, the hardware has been declared a total loss and was replaced.

In this report, we present a rational basis for selecting an approach for removing silicones. We discuss the concept and usefulness of solubility parameters and qualitatively evaluate the solubility of various silicone materials in a variety of organic solvents and cleaners. A visual inspection for the clarity of the resulting solution is used to determine acceptable solvents. Results on the effectiveness of CO₂ jet spray cleaning are also presented.

2. Background

The underlying principle in most cleaning processes is solubility. Solubility describes the tendency of a molecule to become dispersed in a solvent. In simplified terms, molecules disperse into a solvent if the molecule and solvent are "alike." The phrase "like dissolves like" is used frequently. Therefore, a significant need in determining suitable cleaning agents is to understand the ways in which contaminants and solvents are alike.

In soap and detergent chemistry, it has been helpful to classify four types of "soil" and their appropriate cleaners.

- 1. Inorganic—Scale and lime deposits; rust and corrosion; minerals. pH plays a significant role. Acidic cleaners used. Lime-Away[®], citric acid, Acidulate 28[®].
- Biological—Body oil, animal fat; proteins and carbohydrates; mold and yeast; bacterial and animal waste. pH plays a significant role. Alkaline cleaners used (Alconox[®], Liquinox[®], Micro[®]. Most laundry and dishwashing detergents.)
- 3. Organic, Petroleum-based—Synthetic oils, greases, and waxes. No water present. Organic solvents used.
- 4. Combination-Mixtures of the above. Combination cleaners needed.

These soils are largely insoluble in water because they are dissimilar. Soils and water form an incompatible interface that is described as having high surface tension. The surfactants in soaps and detergents help create a more compatible interface between soil and water.

Figure 1 shows a schematic of oily, hydrophobic soil in water. A typical surfactant molecule contains one hydrophobic end and one hydrophilic end. The hydrophobic end is typically a fatty acid while the hydrophilic end can be a carboxylic acid. The fatty acid attaches to and surrounds the oily soil because it is like the soil, while the carboxylic acid points outward toward the water. The surface of the soil particle now appears as an entity that freely mixes with water.

The aerospace industry is largely concerned with the use and removal of organic, petroleum-based materials. The cleanliness of these materials is important because they are likely to outgas in the space environment and cause degrading effects on optical surfaces. In addition, until recently, the surfaces of most aerospace hardware to be cleaned were not compatible with water.* Prior to health and environmental concerns in the 1980s, common aerospace solvents included haloginated fluids such as CFC-113 (Freon TF), tricholorethane, carbon tetrachloride, and methylene chloride. Other common solvents were ketones, such as methyl ethyl ketone and acetone, and isopropyl, ethyl, and methyl alcohols.

^{*} Dishwashing detergents and shampoos, together with mild wiping and rinsing, are now being used to clean telescope mirrors and lenses.



Figure 1. Removal of oily soils in water using a surfactant.

The need to replace haloginated organic solvents and other harmful cleaning chemicals has stimulated efforts to understand the "likeness" of typical aerospace contaminants and organic solvents. In this case, one desires a "likeness" between contaminant and solvent without the need for an intermediate surfactant. One such effort involved finding a replacement for 1,1,1-trichloroethane (commonly used as a dry cleaning solvent), which was used to measure the presence of nonvolatile residue (NVR) on space flight hardware. Solvents that were similar to1,1,1-trichloroethane were identified by examining the Hansen total solubility parameter, δ_{Total} . δ_{Total} consists of three components:

$$\delta_{\text{Total}}^2 = \delta_{\text{P}}^2 + \delta_{\text{D}}^2 + \delta_{\text{HB}}^2$$

where δ_P is the polar parameter, δ_D is the dispersive parameter, and δ_{HB} is the hydrogen bonding parameter. Parameters for common organic solvents are given in Table 1.

Solvent	Dispersive,δ _D (MPa) ^{1/2}	Polar,δ _P (MPa) ^{1/2}	Hydrogen Bonding,δ _{HB} (MPa) ^{1/2}	Total,δ _{Total} (MPa) ^{1/2}	Dipole Moment (Debye)
ethylene giycol	17	11	26	32.9	2.28
methanol	15.1	12.3	22.3	29.6	1.70
diethyl ether	12.4	12.3	23.3	29.1	1.15
2-pyrrolidone	19.4	17.4	11.3	28.4	(3.5)
dimethylsulfoxide	18.4	16.4	10.2	26.7	4.49
ethanol	12.6	11.2	20	26.1	1.69
acetonitrile	10.3	11.1	19.6	24.8	3.92
benzyl alcohol	14.7	12.2	15.6	24.6	
N-methyl-2-pyrrolidone	16.5	10.4	13.5	23.7	2.66
2-propanol (IPA)	14	9.8	16	23.4	1.66
dichloromethane			2-5	É.	1.1.1
acetone	.7 €				$\tilde{\mathcal{L}}(\Omega)$
methyl acetate					
methyl ethyl ketone	1				2.66)
tetrahydrofuran	1.1.1			10° C.	$\{i,j'',i\}$
toluene	÷.,			经济	C: 36
ethyl acetate	$c \in \mathcal{T}_{1}$		~ ~	\$6,0	9 T 6
n-butyl acetate	1.1.28		5.3	67 B	$(\alpha \mathcal{Z}_{p})$
1,1,1-trichloroethane	· · · ·		2.1	ST-T	p 76
carbon tetrachloride	17.8	0.0	0.6	17.8	0.00
cyclohexane	16.5	3.1	0.0	16.8	0.00
heptane	15.3	0.0	0.0	15.3	0.00
hexane	14.9	0.0	0.0	14.9	0.00
Average	15.2	9.3	10.9	22.2	1.9
Median	15.2	10.4	9.9	20.1	1.71

Table 1. Hansen Parameters and Dipole Moments of Common Solvents, Listed by Decreasing δ_{Total}

The Hansen parameters help define quantitatively the similarity of solvents. To find a replacement for 1,1,1-trichlorethane, one sought a solvent with a dominant dispersive parameter, a relatively minor, but finite, polar and hydrogen bonding character, and with δ_{Total} lying in the range of 17–20. As seen in Table 1, the primary candidates (indicated in blue) fell between dichloromethane (methylene chloride) and n-butyl acetate. Several of these candidates were eliminated because of toxicity, flammability, high vapor pressure, high cost, or lack of reasonable availability, leaving ethyl acetate as the chosen solvent. In subsequent testing, it was shown that ethyl acetate removed the same amounts of test contaminants as 1,1,1-trichloroethane. Hansen parameters were therefore capable of indicating the similarity between two solvents and a test contaminant.

In the following sections, the solubilities of silicones in various solvents are examined. A qualitative measure of the solubility is used to select the best cleaning approach.

3. Experimental Approach

Solubility is defined as the concentration of a solute in a solvent. In principle, the best cleaning solvent should obtain the highest concentration of silicone in solution. Therefore, a quantitative measurement of the silicone dissolved into the solvent candidates would be required. In many approaches, however, the saturation level of the solute in the solvent is determined by visual inspection. For the test described in this report, we chose a qualitative observation of turbidity to determine the extent of solubility. We reasoned that this test would be sufficient to determine whether certain solvents could do a better job of removing silicones than what is frequently prescribed, IPA.

Turbidity has been described as a measure of the clarity or "cloudiness" of water. It is used frequently to monitor water quality in environmental studies, often to gauge the growth of algae or to track the presence of clay and silt. As with solubility, the measurement of turbidity can range from simple to complicated. In a simple turbidity study, a patterned wheel, called a Secchi disk, is lowered into a column of water until the patterns can no longer be distinguished. In more involved approaches, well-defined and calibrated light sources and sensors are used to measure light scattered by the particles suspended in the liquid. In each of these cases, turbidity is expressed in either Nephelometric or Jackson Turbdity units (NTU or JTU) that give the fraction of light scattered. However, both NTU and JTU are dimensionless and are sometimes described as being only qualitative. We once again reasoned that a simple visual observation of cloudiness would suffice in order to select an effective solvent for silicones.

Silicone samples were placed in clean glass vials followed by depositing 2–3 mL of candidate solvent. The vial was sealed, shaken, and then photographed. Two series of tests were run. Series I is described in Table 2 and shown in Figure 2. Series I consists of two candidate aqueous cleaners described as combination cleaners/degreasers and one test silicone contaminant, the dimethyl siloxane, E 155 mold release agent.

Series II is described in Table 3 and includes only organic solvents, as well as several more examples of silicone products commonly used in the aerospace industry. The solvents are shown in Figure 3, and the test silicones in Figure 4.

Table 2.	Candidate Solvents and Test Silicone in Series I Solubility Test
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Candidate Solvents	Test Contaminant
Iso-Propyl Alcohol	E155 Silicone Mold Release
Hexane	
Simple Green®	
Orange Citrus Cleaner	



Figure 2. Series I cleaners and solvents: hexane, Nature's Orange, Simple Green, and Iso-propyl alcohol.

Candidate Solvents	Test Silicones	
Iso-Propyl Alcohol	E 155 Mold Release	
Hexane	Dow Corning 705 Vacuum Pump Fluid	
Heptane	Dow Corning High Vacuum Grease	
Toluene	Dow Corning 340 Heat Sink Compound	



Figure 3. Series II Candidate solvents: hexane, isopropyl alcohol, toluene, and heptane.



Figure 4. Series II Test silicone products: E155 mold release, DC high-vacuum grease, DC 705 vacuum-pump fluid, DC 340 heat-sink compound.

We note from Table 1 that heptane is similar to hexane in terms of its component solubility parameters, having only a dispersive value. Heptane, however, has a higher flash point than hexane and would be safer to use if it had efficacy comparable to hexane. Toluene was chosen because it is used as a solvent in the production of silicone resins, suggesting there is some active chemistry between silicone components and toluene. Toluene is also used as a solvent to measure the viscosity of methyl silicone fluids. Carbon tetrachloride is also reported as a solvent used in the production of various silicone compounds. From Table 1, it can be seen that carbon tetrachloride and toluene are also largely dispersive in character, with toluene having the largest polar character.

We present three final notes. First, "hexanes" are often used as a commercial grade solvent. Hexanes are a mixture of hydrocarbon isomers with six C atoms. Hexanes were not tested in this study. In addition, ethyl acetate was not tested because ethyl acetate is viewed as a reference test solvent used in small amounts. It most likely would be considered too expensive if needed for remediation of a large-scale contamination incident and from Table 1, has similarity to toluene.

Finally, this study focuses on common, readily available solvents and cleaners. Dow Corning OS[®] fluids are volatile, low molecular weight methylsiloxanes marketed as precision cleaners and carriers. Since they are silicones themselves, the OS fluids would likely be good silicone cleaners. Reports indicate that some wiping is usually necessary. A ready supply of the OS fluids might be advisable should there be a possibility of silicone contamination.

4. Results

4.1 Series I Test Results

Results from the Series I solubility tests are shown in Figures 5–8. Figures 5 and 6 show the formation of E155 silicone globules floating in the cleaning liquids. These observations indicate that some degree of removal might be possible with either of these cleaning approaches.



Figure 5. E155 silicone globules in citrus cleaner.



Figure 6. E155 silicone globules in Simple Green.

In past applications, cleaning products such as Simple Green were used in conjunction with wiping or other mechanical action. Parts were not dipped. In addition, these products were used in conjunction with copious rinsing with distilled water. In previous experience, therefore, solubility was not the only factor that contributed to cleaning efficacy.

In contrast to Figures 5 and 6, Figure 7 shows immediate and total clarity of the E155 test silicone in hexane. A comparison of the various tests is shown in Figure 8. With further agitation, the silicone globules can be broken up further to form a cloudier, hazier liquid.

To the left of Figure 8 are the samples of E155 in IPA and hexane.



Figure 7. E155 silicone dissolved in hexane.



Figure 8. Turbidity of E155 silicone in (L-R) IPA, hexane, citrus cleaner and Simple Green.

Figures 9 and 10 initially indicate obvious turbidity in a solution of E155 silicone and IPA. However, subsequent observations shown in Figures 11 and 12 suggest that the IPA eventually clarifies.



Figure 9. Turbidity comparison of E155 silicone in IPA (L) and hexane (R). Time, t = 0.



Figure 10. Turbidity of E155 silicone in IPA (L) and hexane (R). t = 5 min.



Figure 11. Turbidity of E155 silicone in IPA (L) and hexane (R). t= 10 min.



Figure 12. Turbidity of E155 silicone in IPA (L) and hexane (R). t = 15 min.

The results from the Series I tests point to immediate solubility of a silicone sample in hexane. Simple Green and citrus cleaners are not as effective, but could be partially effective in removing a silicone. IPA does not appear to be an effective solvent at first. However, over a period of 15 to 20 min, test results indicate that silicones are eventually dissolved. The rate of solubility of silicones in IPA could be slow compared to hexane, or it is possible that the lamp is gradually warming the IPA. In either case, removing silicones with IPA would still be problematic. Since IPA evaporates so

quickly, its use would have to involve a continuous stream or immersion in an IPA bath for about 15 min. Warming of the IPA could also be a problem because of its flammability. Although hexane is also flammable, it appears to dissolve silicones readily at room temperature.

4.2 Series II Test Results

Series II involves evaluating the solubility of several silicone products in hexane, heptane, toluene, and IPA. Simple Green and this citrus cleaner have been eliminated.

4.2.1 E155

Figures 13 and 14 show the initial turbidity of E155 silicone in the Series II solvents, IPA, toluene, heptane, and hexane from two different angles. Immediate and total clarity is observed for toluene, heptane, and hexane. This result is expected for heptane and hexane because of the similarity in their solubility parameters as shown in Table 1. Through a subjective evaluation of clarity, it appears that toluene is an equally effective solvent, even though its solubility parameters are slightly different from those of hexane and heptane. It is possible that toluene has a lower quantitative saturation limit, which is not evaluated in this study.



Figure 13. Turbidity of E155 silicone in IPA (L), toluene, heptane, and hexane (R).



Figure 14. Turbidity of E155 silicone in Series II solvents. IPA (L), heptane, toluene, hexane (R).

4.2.2 DC 705

The turbidity of DC 705 in the Series II solvents is shown in Figure 15. As with E155, good clarity is observed immediately for all solvents except IPA. A time dependence for the solubility of DC 705 in IPA was also observed and is shown in Figures 15–18. High-intensity lighting was not used for this series of photographs, so there is no heating. Near complete dissolution of the DC 705 into IPA took three hours. Note that a larger volume of the solvents is used in the Series II tests.



Figure 15. Turbidity of DC 705 silicone in Series II solvents: IPA (L), toluene, heptane, hexane (R). t = 0.



Figure 16 Turbidity of DC 705 silicone in Series II solvents. T = 60 min.



Figure 17. Turbidity of DC 705 silicone in Seris II solvents. t = 120 min.



Figure 18. Turbidity of DC 705 silicone in Seris II solvents. t = 180 min.

4.2.3 Dow Corning High-Vacuum Grease

The turbidity results for Dow Corning High-Vacuum Grease (DCHVG) in the Series II solvents are shown in Figure 19. DCHVG formed a cloudy solution that did not clarify over a period of several days. Some remaining sample of DCHVG can be seen in the toluene and heptane solutions. While the IPA solution appears clear, it is apparent that the DCHVG sample did not dissolve appreciably. Hexane produced the most complete solution. Silicone greases are known to contain inorganic thickeners that will not be dissolved by organic solvents.

4.2.4 DC 340 Heat-Sink Compound

The turbidity of DC 340 heat-sink compound (DC 340) is shown in Figure 20. Figure 20 shows the solutions with the Series II solvents and also with 1,1,1-trichloroethane. A significant portion of the solid DC 340 remained solid in the IPA and heptane samples. A smaller amount remained as a solid in toluene. The solubility in hexane and 1,1,1-trichloroethane was more complete. After agitation, hexane and 1,1,1-trichlorethane produced cloudy, but uniform wetting of the glass vial surfaces. All samples produced a cloudy, milky solution. Even though the DC 340 sample was poorly dissolved in IPA, the IPA liquid is slightly cloudier than observed for the DCHVG, indicating some slight solubility.

It is difficult to draw strong conclusions from this test because DC 340 contains inert particles by design in order to enhance thermal transfer properties. For this sample, therefore, the cloudy appearance could be due to good solubility of the silicone binder combined with the emulsification of the particles. A simple turbidity test is probably insufficient to determine the effectiveness of solvent cleaning for a more complicated sample such as a heat-sink compound. The heat sink compound is also known to contain inorganic thickeners that will not dissolve in organic solvents.



Figure 19. Turbidity of DC high-vacuum silicone grease in Series II solvents. IPA (L), toluene, heptane, hexane (R).



Figure 20. Turbidity of DC 340 heat sink silicone compound in Series II solvents and 1,1,1-trichloroethane.

5. Silicone Removal by CO₂ Spray

The carbon dioxide (CO_2) jet spray method has been used to remove particulate and molecular film contamination from sensitive surfaces. The method involves mechanical removal in conjunction with solvation of the contaminant in liquid carbon dioxide. If a contaminant is insoluble in liquid CO_2 , it will not be removed. It has been shown that a contaminant must be at least 0.2 mol % soluble in liquid CO_2 for complete removal.* While this is not the case for many silicone oils, attempts have been made to employ CO_2 jet spray to remove them. One such instance is discussed below.

In June 2000, it was discovered that foam packaging used to carry solar cell assemblies had transferred a silicone mold release, used to manufacture the foam, onto the solar cells. The problem was so severe that the contaminated solar cell assemblies (known as "CICs" for coverglass-interconnectcell) were visibly oily, preventing bonding of the CICs to their substrates; this affected an estimated 69,000 cells.

Because of the large number of contaminated CICs, an automated system was thought to be the best method to clean the CICs. CO_2 jet spray cleaning was one of the methods considered. A demonstration was performed with the goal of achieving "visibly clean" surfaces on a small sample of contaminated CICs.

Figure 21 shows two CICs attached to the cleaning stage. The smudges on the solar cell cover-glass in the foreground are silicone oil. The sample shown consists of 2 CICs that together measure 4×6 in.



Figure 21. Contaminated CICs ready for CO_2 spray cleaning. Silicone oil contamination is visible on CIC in the foreground.

^{*} M.M. Hills, "Carbon dioxide jet spray cleaning of molecular contaminants," J. Vac. Sci Technol., A 13(1), Jan/Feb 1995, 30-34.

Figure 22 shows two CICs inside the automated jet spray cleaner. The nozzle is rastered over the surface of the part until the entire area is covered. Figure 23 shows the CICs after removal from the spray booth. The part appears *visibly* clean to the unaided eye.



Figure 22. Cleaning contaminated CICs in automated CO_2 jet spray.



Figure 23. CICs after CO_2 spray cleaning.

Microscopic images of the CICs both before and after CO_2 cleaning are shown in Figures 24 and 25, respectively. Multi-colored interference fringes caused by the presence of oily residue are evident in the image of the CIC prior to cleaning. The fringes are absent after cleaning. Contamination control practices generally require quantifying non-volatile residue on surfaces. Therefore, further study would be necessary to determine whether CO_2 jet spray adequately removed the silicone mold release.



Figure 24. Interference fringes visible in a photomicrograph of silicone-contaminated CIC.



Figure 25. Photomicrograph of the silicone-contaminated CIC after cleaning with CO₂ jet spray.

6. Summary and Conclusions

Because of their wide use, silicones are found in many parts of the aerospace industry. As a result, silicones are often encountered in efforts to recover from processing accidents. Over the years, various organizations have used a variety of cleaners, solvents, and processes in order to remove silicones deposited accidentally. In this effort, we have used solubility and solution turbidity as a figure of merit for finding an effective remover of silicones.

Previously, it was known by some that carbon tetrachloride was an effective solvent for silicones, but this information was apparently not well known among organizations faced with silicone contamination anomalies. These organizations chose commercial degreasers, organic solvents such as acetone and methylene chloride, isopropyl alcohol (IPA), which had mild, but overall unsatisfactory, results. In addition, carbon tetrachloride is now banned due to health and environmental concerns. Knowledge of the solubility parameters would lead one to non-polar, dispersive solvents as a replacement for carbon tetrachloride. These would include hexane, heptane, and other members of the alkane family. In addition, toluene would be a candidate since it participates as a solvent in the manufacture of silicone resins.

A small selection of these solvents—hexane, toluene, heptane, and IPA—was developed to test their solubility with various silicone products. Hexane, together with its alkane relative, heptane, and toluene, were found to be good solvents for silicone fluids. The solutions formed had low turbidity. None of the solvents were found to be good dissolvers of silicone greases or pastes, however. Some solid or gelatinous residue remained, and the resulting solutions were cloudy. IPA was the poorest in dissolving silicone greases, although the IPA liquid remained the clearest because such small amounts of the solid dissolved into solution.

Initially, IPA formed turbid solutions with "pure" silicone fluids. Surprisingly, the solutions were seen to clarify over time. Heat and increased solvent volume seemed to enhance the solubility. This result indicates that there is some solubility of silicones in IPA, given enough time, and perhaps some mild heating below the IPA flashpoint. However, because IPA evaporates so quickly, IPA would only be useful if available as a continuous spray or as a bath into which a part or component could be immersed for an extended time. This is a concept well worth investigating since hexane or heptane, while more aggressive, could damage sensitive surfaces in the process of removing silicone contamination.

Where large-scale cleaning is involved, or if there are many parts to be cleaned, an automated cleaning process would be desirable. CO_2 jet spray was studied as a means for cleaning large numbers of solar cell CICs contaminated with a silicone mold release. CO_2 jet spray was successful in achieving "visibly clean" CIC surfaces, but past literature indicates that silicones are only partially soluble in CO_2 and that some remaining residue on the surfaces is likely.

LABORATORY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security programs, specializing in advanced military space systems. The Corporation's Laboratory Operations supports the effective and timely development and operation of national security systems through scientific research and the application of advanced technology. Vital to the success of the Corporation is the technical staff's wide-ranging expertise and its ability to stay abreast of new technological developments and program support issues associated with rapidly evolving space systems. Contributing capabilities are provided by these individual organizations:

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Space Materials Laboratory: Evaluation and characterizations of new materials and processing techniques: metals, alloys, ceramics, polymers, thin films, and composites; development of advanced deposition processes; nondestructive evaluation, component failure analysis and reliability; structural mechanics, fracture mechanics, and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures; launch vehicle fluid mechanics, heat transfer and flight dynamics; aerothermodynamics; chemical and electric propulsion; environmental chemistry; combustion processes; space environment effects on materials, hardening and vulnerability assessment; contamination, thermal and structural control; lubrication and surface phenomena.

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