MATERIALS ENGINEERING INVESTIGATION
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NTSB Grease Evaluation

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1.0 Abstract

A comprehensive study involving the examination, experimental testing, and analysis of lubricating grease was performed. Chemical analysis of grease samples taken from the accident aircraft revealed the presence of at least two brands of lubricating grease. Microscopic analysis of those samples revealed the presence of aluminum bronze wear particulates distributed within the grease sample. Physical testing of virgin samples of the two brands of greases were performed using standard tests according to ASTM and Federal Test Methods. These tests were used to examine the effects of intermixing of these greases on both the physical and chemical characteristics of the greases.

2.0 Introduction

A comprehensive study of lubricating grease was performed to support the National Transportation Safety Board's investigation of the Alaska Airlines flight 261 accident. The work performed by the Aerospace Materials Division (AMD) focused on two areas. The first was an analysis of in-service grease samples recovered from the accident aircraft. The goal was to identify the type of grease and to identify any solid particulate debris present in the grease. Since it was determined from maintenance records that the jackscrew was serviced with two different types of grease, the second focus of the investigation was to determine the physical and chemical properties of the two virgin greases and blends of the two. The two greases were Aeroshell 33 manufactured by Equilon Enterprises and Mobilgrease 28 manufactured by Exxon-Mobil.

3.0 Experimental and Analytical Techniques

This section describes the procedures and techniques used in the testing, examination, and analysis of the variety of greases and grease samples covered in this report.

3.1 In service Grease Samples

The examination and analysis of several grease samples taken from both the accident aircraft and several similar aircraft have been performed[1]. Chemical analysis to identify the types of greases present was performed using Fourier Transform Infrared Spectroscopy (FTIR). Microscopic analysis of the grease samples for the detection and identification of foreign solid materials was performed using Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM/EDS).

Several samples of grease taken from in-service aircraft were analyzed by AMD, with the assistance of the Materials Laboratory at NADEP Jacksonville, and the Materials

[1] Samples were provided to AMD in small volumes (typically less than 1cc) in glass sample containers by NTSB.

Laboratory at NADEP Cherry Point. A list of participants in this study is found in the "Acknowledgements" section at the end of the report.

3.1.1 Fourier Transform Infrared Spectroscopy

Analysis was performed in accordance with the AMD Quality Plan and followed the Technical Procedure "FT-Infrared Analysis via Microscopic FTIR". The instrument used was a Nicolet Nic-Plan microscope attached to a Nicolet Magna 550. Each sample of grease was analyzed neat, using a diamond compression cell placed on the stage of the microscope. The diamond compression cell allows for analysis without mixing the sample into a pellet or mull.

3.1.2 Scanning Electron Microscopy/Energy Dispersive X-Ray

Grease samples were examined and photographed with optical microscopy. High magnification imaging was performed in a JEOL JSM5800LV scanning electron microscope (SEM) operated at 20 KV in the low-vacuum mode (which allows examination of nonconductive samples without the application of a gold or carbon coating). An Edax DxPrime Energy dispersive X-ray spectrometer system (EDS) was used to identify non-organic particles within the grease. Large particles that coated the outside of the grease clumps were identified using a Rigaku System D/2000 X-ray diffractometer (XRD) operated at 40kV and 30 mA.

3.2 Virgin Grease Testing

Virgin samples of Mobilgrease 28 and Aeroshell 33 were evaluated for compatibility in accordance with ASTM D-6185, Standard Practice for Evaluating Compatibility of Binary Mixtures of Lubricating Grease. The standard practice evaluates the properties of the mixtures relative to the neat greases that comprise the mixture. In general, the measured physical property of the mixed grease must fall within the range of measurements of the two neat greases for the two greases to be considered compatible. Samples tested included 100% Mobil grease 28, 100% Aeroshell 33, and 50/50, 90/10, and 75/25 mixtures of the two greases. The 50/50 ratio simulates the condition when one grease is added to a bearing, which contains another grease. The 90/10 and 10/90 ratios simulate attempts to flush out one grease with another. The tests performed were dropping point by Test Method D2265, and shear stability by Test Method D217 (this method covers 100,000 stroke worked penetration and storage stability at elevated temperature).

The effects of submersion in seawater were examined by immersing virgin greases in seawater. [2] Test samples were 100% Mobil grease 28, 100% Aeroshell 33, and a 50/50 mixture of the two. The grease was weighed into a glass jar and filled with

[2] Because the accident aircraft had crashed into the Pacific Ocean the grease samples recovered from the wreckage were subjected to a prolonged exposure to seawater.

seawater recovered from the accident site. The jars were capped and stored in a refrigerator at 40°F for two weeks.

Corrosion screening tests were performed according to ASTM Method D 4048 and Federal Standard 791 Method 5309.5. Each method uses copper as the test metal. For this investigation, aluminum bronze was also tested for comparison. Coupons of copper were immersed in grease and placed in a 100ºC oven for 24 hrs. They are then compared to ASTM Copper Strip Corrosion Standards and rated according to Table 1 in ASTM D 4048.

4.0 Results of Testing and Analysis

This section describes the results of the testing, examination, and analysis of the variety of greases and grease samples covered in this report.

4.1 In-Service Grease Samples

Several samples of grease were taken from the jackscrews of both the accident aircraft, and other similar aircraft. Table 1 identifies these samples by NTSB sample number, the aircraft tail number, and the location from which the sample was taken.

Table 1. Sample Identification

NTSB Sample Number	Sample Description
59	N963AS Lower end of screw
53	N963AS Zerk Fitting orifice
56	N963AS Gimbal Nut
58	N963AS Lower Stop
61	N963AS A/C strut below jackscrew
31	N981 AS Below screw/nut junction
2	NWA Acme Screw
5	N982AS Acme Screw
8	Delta DCA 0009 Acme Screw

4.1.1 Chemical Analysis by FTIR

Samples #0316002 (Virgin Mobil Grease 28) and #0316003 (Virgin Aeroshell Grease 33) were analyzed via microscopic FTIR and the resultant spectra were saved in the FTIR software's spectra library to be used as standards for comparison. Additionally a 50/50 mix of the two greases, 10/90 and 90/10 mixes of the two greases, each of the virgin greases after exposure to seawater-retrieved from the accident site, and samples of the greases and mixes after they had been exposed to a four ball wear test were also prepared and the resultant spectra were stored in the same manner and with the same intent.

The following table summarizes the results of the samples that were observed and analyzed using FTIR. Details of this analysis are found in the Analytical Laboratory report number 031600.

Table 2. FTIR Analytical Results

AMD Sample Number	NTSB Sample Number	Visual Observations	Substance Identification
0316001	59	Sand covered, dark greasy globs with many imbedded fibers	Mix of Aeroshell 33 and Mobil 28
1025001	53	Tiny black and white particles of very dry grease, a few small orange particles also evident	(B)-Mix of Aeroshell 33 and Mobile 28 (10:90) & (G)-Mix of Aeroshell33 and Mobile 28 (50:50)
1025005	56	Dark greasy globs, reddish-brown tint, still greasy	Mobil 28
1025007	58	Black-yellow grease clumps with fibers and particle contamination imbedded in sample, losing its greasy consistency	Mix of Aeroshell 33 and Mobil 28 (10:90)

1025009	61	Black-red streaking to yellow, another area appears black-green, sample retains greasy consistency	Unable to make identification at this time
1025003	31	Dark greasy globs, green-brown in color, many metallic flakes observed in sample	Aeroshell 33
10250011	2	Brown-Black grease with red streaks	Mobil 28
10250013	5	Dark grease with light green tint, loaded with particulate contamination	Mix of Aeroshell 33 and Mobil 28 (90:10)
10250015	8	Very oily dark black grease with red spots, loaded with tiny particles	Unable to make identification at this time

4.1.2 Scanning Electron Microscopy / Energy Dispersive X-ray Spectrometry, X-ray Fluorescence, and X-ray Diffraction Analysis

Scanning electron microscope/energy dispersive X-ray spectroscopy (SEM/EDS) analysis of the particulate contaminants was performed at the Materials Laboratory at NADEP Cherry Point (see Appendix A) and at the Aerospace Materials Laboratory (AMD) at Patuxent River. Results were consistent at both laboratories. X-ray fluorescence was used at the Materials Laboratory at NADEP Jacksonville (see Appendix B), and yielded the same results. A summary of the AMD results are given below. Details of this analysis are found in the Microstructural Laboratory Particulate Analysis report.

<u>Samples from the Accident Aircraft N963AS</u>

Visual examination of the grease sample 59 showed the presence of several discrete black clumps and a scattering of small particles contained within a plastic sample holder. On examination under a stereo zoom optical microscope, the clumps appeared to be coated with a combination of fibers and translucent particles that had the appearance of sand. The fibers that were present on the surface of the clumps appeared similar to those that composed the filter paper, which lined the sample container. A portion of grease removed from Sample 59 was cut to reveal an interior surface, and placed in the SEM for examination. Backscattered electron images, which are sensitive to sample density, showing dense material as bright, revealed a large number of dense (metallic) particles in the grease, varying in number and size. The smallest of the particles were sub-micrometer in size. Elemental analysis with EDS showed that these particles were of a relatively uniform composition, consistent with C95500 brass (AMS 4880).

Morphologically the particles were generally flat flakes. The flakes show several cracks and parallel scoring. These types of particles have characteristics consistent with that of particulate generated by a sliding wear mechanism.

XRD was used to determine that the large translucent particles were sand.

The remaining four samples from the accident aircraft (53,56,58,60) were all dark in color, but there were some distinguishing features among them. Sample 56 appeared distinctly redder in tint than the other three. Sample 58 appeared to be the driest looking of the four but all were less dry in appearance than the original sample (59). There were no fibers or translucent particles on the grease surfaces.

Sample 56 contained metallic particles, but they were distributed non-uniformly. Bands of high particle density could be seen along with regions containing few particles. Sample 58 appeared similar to 56 with more pronounced banding of the metallic particles. Sample 61 contained uniformly distributed particles.

Samples from Alaska Airlines Aircraft N981AS and N982AS

Sample 31 contained a uniform high density of metallic particles similar to sample 59. EDS showed that the particles were consistent with aluminum bronze.

SEM examination of Sample 5 revealed that the sample contained many particles, with a particle density approaching that seen in the samples taken from the accident aircraft. Distribution of particles was fairly uniform. Particles extracted from the grease were in the form of flakes as long as two millimeters in length. EDS revealed that these particles were consistent with aluminum bronze alloy C95500. Some of the flakes appeared to have areas on the surface that were slightly darker and more reddish in color than others. EDS showed that the dark flakes were the result of oxidation.

<u>Samples from Delta and Northwest Airlines Aircraft</u>

Grease samples from Northwest and Delta appeared less dry than the accident aircraft sample, did not clump and were not coated with fibers or translucent particles. All were dark in color; sample 2 had a slight reddish tint.

Sample 8 contained a non-uniform distribution of metallic particles.

Sample 2 contained some metallic particles, but less than the Delta and Alaska Airlines samples. Particles extracted from the grease were in the form of flakes and long shavings. EDS analysis revealed that these particles were of two distinct compositions: aluminum bronze and an aluminum alloy. The source of the aluminum bronze can be assumed to be the jackscrew nut. The source of the aluminum is unknown. In this sample the largest particles were flakes of the aluminum alloy.

4.2 Virgin Grease Testing

4.2.1 Grease Compatibility

ASTM D-6185 is a standard test for evaluating the compatibility of binary mixtures of lubricating greases. Three grease characteristics were evaluated in this method including dropping point, high temperature storage stability, and shear stability. The two greases were blended into specific ratios of 50/50, 90/10 and 75/25.

Compatibility testing of several mixtures of virgin samples of Mobilgrease 28 and Aeroshell 33 indicated that these two greases are incompatible at the 90/10 and 10/90 ratios. This was evidenced by the storage and shear stability test results, however the results were such that the change does not indicate a significant failure of the grease mixture.[3]

4.2.2 Cone Penetration of Lubricating Greases ASTM D217

Cone penetration evaluates the consistency of lubricating greases. The results of the worked penetration, shear stability, and high temperature storage stability are shown below. High temperature storage stability and shear stability tests simulate

[3] Mobilgrease 28 contains a clay thickener and a synthetic hydrocarbon base. On the other hand, Aeroshell 33 is composed of synthetic base oil with a lithium complex thickener. Greases that contain different types of thickeners can be incompatible. For this reason, equipment manufacturers usually recommend that grease be completely cleaned from equipment before new grease is added.

the effects of storage/aging and mechanical shear on the consistency of lubricating greases.

Table 3. Cone Penetration of Virgin Greases and Mixtures

% Mobilgrease 28	100	90	75	50	25	10	0
% Aeroshell 33	0	10	25	50	75	90	100
Penetration, (1/10 mm penetration) ASTM D-217							
Initial (Worked)	312	308	310	298	304	283	282
70 hrs. @ 120 ^oC (storage stability)	296	291	295	293	309	320	304
Change from initial	-16	-17	-15	-5	+5	37	22
Shear Stability	305	335	310	306	310	275	294
Change from initial	-7	+27	0	+8	+6	-8	+12
Compatibility	N/A	Incompatible*	Compatible	Compatible	Compatible	Incompatible *	N/A

* If the change in penetration of the mixture is greater than that of the constituent greases by an amount greater than the repeatability of the test method (7 units)[4], the greases are incompatible.

4.2.3 Dropping Point ASTM D2265

The dropping point of a grease is the temperature at which the first drop of material falls from the test cup. When greases are mixed, the dropping point can be reduced to the point where separated oil will run out of bearings or other equipment at elevated temperatures. Dropping point results are shown below.

Table 4. Dropping Point of Virgin Greases and Mixtures

% Mobilgrease 28	100	90	75	50	25	10	0
% Aeroshell 33	0	10	25	50	75	90	100
Dropping Point, ^oC, ASTM D-2265	385+	385+	300+	252	213+	214	219
Compatibility	Na	Compatible	Compatible	Compatible	Compatible	Borderline **	Na

** If the dropping point of the mixture is less than the lower of the constituent greases by an amount equal to or less than the repeatability of the test method (8 units), the greases are borderline compatible.

4.2.4 Oil Separation ASTM D 6184

Oil separation is used to determine the tendency of the grease mixtures to separate oil at an elevated temperature. When a grease separates oil, the ability of the grease to function as designed is impaired. This test is not intended to predict oil separation under service conditions. No unusual results were observed during these tests. The results are listed in Table 5.

[4] Per ASTM D-6185

Table 5- Oil Separation of Virgin Greases and Mixtures

% Mobil Grease 28	100	90	50	10	0
% Aeroshell	0	10	50	90	100
Oil Separation (%)	0.9	1.4	2.3	2.3	3.5

Note: Specification limits are 2-8%(at 177ºC) for Mil-PRF-81322 and 5% max for Mil-PRF-23827

4.2.5 Salt Water Exposure

After two weeks immersion in seawater, the virgin samples of Mobil Grease 28, Aeroshell 33, and the 50/50 mixture exhibited only slight color changes, with no other adverse effects noted. Each sample remained adhered to the glass jar and in one clump.

4.2.6 Corrosion Screening Tests on Copper and Aluminum Bronze Alloy

Copper and aluminum bronze coupons were tested according to ASTM Method D 4048 and Federal Standard 791 Method 5309.5. The test measures the tendency of grease to corrode copper under static conditions. No correlation has been established with actual field service. The test method specifies hard-temper, cold-finished copper of 99.9+% purity. Aluminum bronze was included in the test for comparison. Coupons that failed at the interface of the grease and air exhibited a dark stain along the grease line. The 50/50 mixture expanded in the oven, causing pockets to form in the grease. When removed, the coupons showed dark spots consistent with the formation of such pockets. The results show a slight discoloration of some samples. The results are shown in Table 6.

Table 6 Corrosion Screening Tests

Test Number	Material	Grease Type	Test Method	Results
1	Copper	Mobilgrease 28	FTM 791	Pass 1 a
2	Copper	Mobilgrease 28	ASTM 4048	Pass 1 a
3	Aluminum Bronze	Mobilgrease 28	FTM 791	1 a
4	Aluminum Bronze	Mobilgrease 28	ASTM 4048	1 a
5	Copper	Aeroshell 33	FTM 791	Fail 2 e- Dark stain at interface
6	Copper	Aeroshell 33	ASTM 4048	Pass 1 a
7	Aluminum Bronze	Aeroshell 33	FTM 791	2 e – Dark stain at interface
8	Aluminum Bronze	Aeroshell 33	ASTM 4048	1 a
9	Aluminum Bronze	90% Mobilgrease 28 /10% Aeroshell 33	FTM 791	2 e slight stain
10	Aluminum Bronze	90% Mobilgrease 28 /10% Aeroshell 33	ASTM 4048	2 e

11	Aluminum Bronze	50/50 Mix	FTM 791	2 e Dark spots
12	Aluminum Bronze	50/50 Mix	ASTM 4048	2 e Dark spots
13	Aluminum Bronze	90% Aeroshell 33 /10% Mobilgrease 28	FTM 791	1 b
14	Aluminum Bronze	90% Aeroshell 33 /10% Mobilgrease 28	ASTM 4048	1 b

5.0 Conclusions

5.1 Accident Aircraft Grease Samples

Analysis of the samples from the accident aircraft indicates the presence of Aeroshell 33. The results also indicate the presence of Mobilgrease 28. While the organic analysis is not completely conclusive, it appears that both greases are present.

Particulate contamination was in two forms. The exterior surfaces of the grease clumps were coated with sand, presumably from the crash site, and fibers that may have come from the recovery operation. This material was not present prior to the crash, or it would have been mixed throughout the grease. The interiors of the grease clumps were relatively free of sand and fibers, but were dense with particles of aluminum bronze. These bronze particles were relatively uniform in composition, and almost certainly originated from a single source. Their composition is consistent with C95500 aluminum bronze. The microscopic flat flakes and shavings are consistent with wear debris of a piece of C95500 aluminum bronze. No other wear debris was found, and no further analysis is warranted.

5.2 Virgin Grease Testing

High temperature storage stability and shear stability testing indicated that there was an incompatibility in the 90/10 and 10/90 mixtures. No incompatibility was found at the 50/50 or 75/25 ratios. Although this is unusual, F.S. Meade documented a case where two greases were compatible at the 50/50 ratio, but incompatible at the more dilute ratios (see ASTM D-6185, footnote 7 and Appendix X1).

Evaluation of the dropping points of the mixtures of the two greases indicated that the 10% Mobilgrease 28/90% Aeroshell 33 ratio was borderline compatible. The other mixtures were compatible.

Although the greases were determined to be incompatible according to the procedure, no significant physical changes were observed. The results that were incompatible do not indicate a significant failure of the grease mixture.

References

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2. *ASM Handbook,* Volume 2, Properties and Selection: Nonferrous Alloys and Special-Purpose Materials, ASM International, Metals Park, Ohio, 1995, p. 385.

3. ASTM D-217 – "Test Methods for Cone Penetration of Lubricating Grease"

4. ASTM D-2265 – "Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range"

5. ASTM D-6185 – "Standard Practice for Evaluating Compatibility of Binary Mixtures of Lubricating Greases"

6. FED-STD-791, Method 3467.1 – "Storage Stability of Lubricating Grease"

7. MIL-G-7711 – "Grease, Aircraft, General Purpose"

8. MIL-PRF-23827 – "Grease, Aircraft, Extreme Temperature"

9. MIL-PRF-81322 – "Grease, Aircraft, General Purpose, Wide Temperature Range"

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