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Designation: D8182 – 18

Standard Test Method for Alloy Classification of Wear Debris using Laser-Induced Breakdown Spectroscopy (LIBS)[1]

This standard is issued under the fixed designation D8182; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a means for quantitative determination of wear debris found in in-service lubricants by laser-induced breakdown spectroscopy (LIBS). LIBS is an analytical technology that uses short laser pulses to create micro hot-plasma ablation of a material and then employs spectroscopic tools for analysis.[2]

1.2 This method covers the means for alloy classification and sizing of wear debris. Wear debris sources can include, but are not limited to: *(1)* chip collector and chip detector devices, *(2)* filters, *(3)* ferrograms, and *(4)* loose particles. The 23 tested alloys and metals included in the default material library of the instrument are listed in Table 1.

1.3 The method for alloy classification and sizing of wear debris is not limited to the list of alloys in Table 1. The instrument has the capability of including additional alloys and metals as required.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D7669 Guide for Practical Lubricant Condition Data Trend Analysis

D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

3. Terminology

3.1 *Definitions:*

3.1.1 *alloy, n*—unique composition of two or more metals that has one or more of the metals treated or processed in a special way to confer enhanced performance characteristics on the resulting material.

3.1.2 *debris, n*—solid particulate matter introduced to lubricant (or machinery/equipment fluid) through contamination or detached from a surface due to wear, corrosion, or erosion process.

3.1.3 *in-service oil, n*—lubricating oil that is present in a machine that has been at operating temperature for at least one hour.

3.1.4 *wear, n*—damage to a solid surface, usually involving progressive loss or displacement of material, due to relative motion between that surface and a contacting substance or substances.

3.1.5 *wear debris, n*—particles that have become detached in wear or erosion processes.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *alloy classification, n*—the automated process that uses the LIBS technology in conjunction with an artificial neural network (ANN) to determine the specific alloy or alloy grouping for a given sample.

3.2.2 *alloy grouping, n—within a given LIBS training set*, when two or more alloys are significantly similar in elemental

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[2] Hill, R., Lawrence, R., Toms, A.; "A New Approach to Elemental and Wear Debris Analysis," STLE, Las Vegas, NV, 2016.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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TABLE 1 List of 23 Alloys and Metals

Alloys
1010 Carbon Steel
17-4PH Stainless Steel
300 Series Stainless Steel (316, 321, 347)
400 Series Stainless Steel (416, 410)
52100 / 4130 Alloy Steel
9310 / 4340 Alloy Steel
A286 Stainless Steel
Aluminum 2024
Aluminum 6061
Aluminum 7075
Brass
Copper
Inconel 718
M50 NiL Steel
M50 Steel
Silver
Titanium 6Al-2Sn-4Zr-6Mo
Titanium 6Al-4V

composition, the alloy classification capabilities of the instrument may not be able to distinguish them as separate or individual alloys; when this occurs, such alloys are combined under a more general classification referred to as an "alloy grouping" (for example, 52100 and 4130 may be combined within a single alloy grouping of "52100/4130").

3.2.3 *laser-induced breakdown spectroscopy (LIBS), n*—a rapid chemical analysis technology that uses a short laser pulse to create a micro-plasma on the sample surface.

3.2.4 *material library, n*—the material library is the instrument's collection of alloy and metal classification outputs; the material library is a subset of the training set and may contain individual alloys or alloy groupings.

3.2.5 *sample, n*—test specimen or a collection of wear debris that is first placed on a sample patch and then inserted into the instrument for analysis.

3.2.6 *sample patch, n*—customized plastic insert used for LIBS analysis.

3.2.6.1 *Discussion*—A given sample patch contains a 0.5 in. by 0.5 in. clear window with semi-permanent adhesive for wear debris retention. Sample patches are populated with samples of wear debris and then inserted into the instrument for analysis.

3.2.7 *training set, n*—the collection of certified alloy and metal samples and the LIBS spectra acquired from these alloys, which are used to define and configure the instrument's material library.

4. Summary of Test Method

4.1 Wear debris is extracted from a chip detector or other source and placed onto a transparent adhesive sample patch, Fig. 1. The patch is transferred to an instrument that uses a combination of LIBS, an artificial neural network (ANN) and digital imaging technologies to determine specific alloy classification and size. To determine the size of a given piece of wear debris, the transparent sample patch is back lit to create a silhouette of the wear debris and a high-resolution image is processed using an image binarization and processing algorithm. In the case of large pieces of wear debris, the software allows for each particle to be analyzed in several locations. This option allows the instrument to determine if the image of the wear debris is one piece or several overlapping pieces of different alloys or metals.

FIG. 1 Transparent Adhesive Sample Patch with Chip Debris

4.2 The LIBS instrument uses short laser pulses to create micro hot-plasma ablation on the surface of the wear debris and then employs spectroscopic tools to analyze the materials composition. A plasma field from the ablation spot emits various wavelengths of light unique to the elements present in the sample being analyzed. A spectrometer is used to determine intensities of each element present in the sample and an artificial neural network (ANN) analyses this data to output the specific alloy classification for each individual piece of wear debris analyzed.

4.2.1 Alloy classification is determined based on the composition of the wear debris under analysis.

4.3 Instrument calibration is supplied by the manufacturer via measurement of the following, and is maintained in service as defined in 13.2:

4.3.1 An alloy training set comprised of all of the alloys required in the instrument's material library is used as references to calibrate the instrument for alloy classification. Where possible, certified alloy or metals are sourced from multiple vendors and accompanied by third party certification. A large number of spectra are collected over a range of intensities and this aggregate collection of data is used to calibrate the instrument's ANN.

4.3.2 For image calibration, a US Air Force 1951 Resolution Target is used to configure the imaging optics at the factory. For field operation, a field calibration standard is used to establish the ratio between image pixel and wear debris size.

5. Significance and Use

5.1 In many cases, equipment failure modes are identified by wear debris that is not captured in used lubricating oil samples but captured on chip detectors, filters or by other means. Users of this technique include, but are not limited to, original equipment manufacturers (OEMs), commercial airlines, civil aerospace operators, maintenance repair and overhaul (MRO) facilities, and military maintenance personnel.

6. Interferences

6.1 For optimum wear debris classification and instrument performance, the following guidelines should be followed to reduce interferences.

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6.1.1 Residual oil on the wear debris may weaken the signal intensity. An isopropyl alcohol rinse of the debris is recommended prior to applying the wear debris to the sample patch. Applying a drop or two of isopropyl alcohol to the sample patch, after the wear debris is applied, is allowed.

6.1.2 When preparing the sample patch for analysis, concentrated 'clumps' of wear debris should be avoided. Overlapping debris may hinder proper identification of some particles. If needed, excessive debris all from one source can be placed on multiple patches.

6.1.3 When preparing the sample patch for analysis, wear debris should lay as flat as possible on the sample patch for proper identification.

6.1.4 Avoid placing wear debris thicker than 0.5 mm on a sample patch. Wear debris thicker than 0.5 mm should be broken into smaller pieces for proper identification.

6.1.5 Alloys not in the material library will be classified as "unclassified" or if their elemental composition is similar to an alloy in the library, they may be incorrectly classified.

6.1.6 The operation, maintenance, service and all related activities regarding the instrument shall be performed in accordance with the manufacturer's manual and technical specification.

7. Apparatus

7.1 *LIBS Instrument.*[4]

7.2 *Sample Tray,* to hold the transparent adhesive sample patch containing the wear debris sample during analysis.

8. Reagents and Materials

8.1 Transparent adhesive sample patches.

8.2 Optional patch preparation tools and materials (tweezers, probe set, wash bottle, isopropyl alcohol).

8.3 Standardization standard that allows for the standardization procedure to be completed verifying the instrument is standardized.

8.4 Calibration standard that allows for the calibration procedure to be completed ensuring instrument calibration.

9. Hazards

9.1 Potential hazards arising from the use of a laser have been mitigated by the design and manufacturing process. The instrument is designated as a Class 1 laser product and complies with US FDA performance standards for laser products except for deviations pursuant to Laser Notice No. 50, dated June 24, 2007. Only manufacturer authorized technicians can complete laser service on the instrument. Manufacturer recommended safety procedures must be followed during instrument service.

[4] The sole source of supply of the apparatus known to the committee at this time is GasTOPS, Ltd., Polytek St., Ottawa, Ontario K1J 9J3, Canada. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

10. Sampling, Test Specimens, and Test Units

10.1 For alloy classification, wear debris particles with a minimum dimension larger than 70 µm are classified. Particles smaller than these dimensions are not classified.

10.2 Test specimens and wear debris samples should be no thicker than 0.5 mm. Any sample that is thicker than 0.5 mm should be broken into smaller pieces.

10.3 Residual oil on the wear debris should be removed prior to applying the wear debris to the sample patch. An isopropyl alcohol rinse of the debris is recommended.

10.4 For proper identification, test specimens and wear debris samples must be alloys or metals included in the material library and training set.

10.5 Test specimens and wear debris samples should be flat for proper identification.

11. Preparation of Apparatus

11.1 Refer to the manufacturer's manual for the mechanical setup and power up procedures.

11.2 Ensure that no alerts, which indicate a fault with the measurement hardware or software, are generated during powerup.

12. Conditioning

12.1 The instrument self-conditions upon start-up. The instrument will initiate and automatically notify the operator when ready.

13. Calibration and Standardization

13.1 *Standardization*—In-service standardization is completed by the operator in order to verify the status of the instrument. Standardization is completed through the use of an automated standardization process using manufacturer provided standardization standard. The standardization procedure should be completed once each day prior to use of the instrument. Refer to the manufacturer's manual for the standardization procedure.

13.2 *Calibration*—The instrument is factory-calibrated upon receipt. In-service calibration, if required, is completed by the operator through the use of an automated calibration process using manufacturer provided calibration standard. Refer to the manufacturer's manual for the calibration procedure.

14. Analysis Procedures

14.1 The instrument shall be used, operated and maintained in accordance with the instrument's manufacturer's requirements, manual and technical specifications. The following is a high-level summary of the most critical procedures required to ensure optimal performance from the instrument. Refer to the manufacturer's manual for all other procedures including transportation, maintenance and service.

14.1.1 *Sample Patch Preparation*—There are several potential sources of wear debris. The process of transferring wear debris from the collection source to the sample patch largely depends on how the debris was collected from the equipment.

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The following procedure defines the recommended process of transferring wear debris while considering the cautions outlined in Section 6.

14.1.1.1 *Cleaning Wear Debris from a Chip Detector, Chip Collector, or Magnetic Plug*—Remove the collection device from the equipment and submerge the portion of the device that contains the wear debris in an alcohol bath. Keep the wear debris submerged and continuously rotate the collection device within the alcohol bath for no less than 10 s. If an alcohol bath is not available, then vigorously spray the wear debris and the collection device with isopropyl alcohol for no less than 10 s. If oil residue remains on the wear debris, complete an additional alcohol rinse on the wear debris and allow the wear debris to fully dry. If necessary, repeat this step until no oil residue remains on the wear debris.

14.1.1.2 *Cleaning Wear Debris from an Alternative Source*—If debris is from an alternative source such as a filter, transfer all wear debris from the collection device to an intermediate receptacle (such as a petri dish) for the alcohol rinse.

14.1.1.3 Take a sample patch and peel/remove the protective liner from the top surface of the patch. This will expose the transparent adhesive used to retain the wear debris. Take all precautions to limit the amount of contamination (dust, dirt, etc.) that can now adhere itself to this exposed area.

14.1.1.4 Transfer the clean and dry wear debris from the chip collector to the sample patch by pressing onto the transparent adhesive. If debris is from an alternative source, laboratory grade tweezers and probes should be used for the transfer process and each piece of wear debris should be placed on the sample patch following the cautions outlined in Section 6. It is recommended that wear debris is transferred to the sample patch one at a time. Note that, multiple sample patches can be used for this step, if needed.

14.1.1.5 If the wear debris will not be immediately analyzed, replace the protective liner over the wear debris and transparent adhesive of the sample patch. This will protect the sample from further contamination until the analysis can be completed. Ensure the protective liner is removed prior to running an analysis in the instrument.

14.1.2 *Sample Patch Loading (insertion into the instrument)*—Once a sample patch has been prepared in accordance with Sections 6 and 14.1.1, it can be inserted into the instrument. Refer to the manufacturer's manual for the sample insertion procedure.

14.1.3 Once a sample patch has been prepared and inserted into the instrument in accordance with Sections 6, 14.1.1 and 14.1.1.5, the following procedure should be followed in order to run an analysis on the instrument:

14.1.3.1 Using the instrument's integrated touchscreen, enter all of the mandatory sample identification information and start the automated LIBS analysis. Refer to the manufacturer's manual for the analysis procedure.

14.1.3.2 Upon analysis completion, the alloy classification, area (in microns squared) and location of each of the analyzed wear debris are displayed on the instrument's screen and are automatically saved in the instrument's database.

14.1.3.3 The instrument may be preset to analyze each wear debris in several locations/sections.

15. Calculation or Interpretation of Results

15.1 The wear debris alloy classification, location on the patch and wear debris area (in microns squared) results are automatically determined by the instrument through its internal alloy and image processing calibrations and are displayed to the operator.

15.2 Any resulting classifications output from the instrument are interpreted as nearest matches. Note that if machine recommendations are included, results may give a clear indication that the machinery should not be operated; however, this does not necessarily mean that in the absence of such cautionary indications, that the machine is safe for continued use.

15.3 *Trending and Alarm Limits*—According to the particular procedures employed by the operator, the device will acquire, report and log the results of this test method. Based on the operator's knowledge of their lubricant and machinery, the user may monitor the trending behavior of the reported wear debris alloy classification and its size. Guidance on performing such trending analysis and setting alarm limits on in-service lubricants and machinery is provided in Guides D7669 and D7720.

16. Report

16.1 Once the instrument has completed its automated analysis on the sample, results are immediately displayed on the screen and are automatically written to a database file which may be utilized for further analysis. Furthermore, PDF reports are automatically generated for each analyzed sample patch and can be referenced at any time.

16.2 Analyzed wear debris is reported by the specific alloy classification.

16.3 The area of each wear debris particle is reported in microns squared (μm^2). Units of measure are configurable.

16.4 An image of the sample patch is included in the report.

17. Precision and Bias

17.1 A preliminary precision study, based on a single operator applying the test method, has been performed using a range of particles of different alloys and sizes. Samples were prepared by placing wear debris of various sizes and shapes of each alloy on the transparent adhesive sample patch. Each patch was placed into the LIBS instrument and analyzed twice for alloy identification and wear debris sizing results. A temporary precision statement was made using the methods described in Practice E177. The repeatability standard deviation from a single operator is displayed in Table 2. Since

TABLE 2 Preliminary Precision for Wear Debris Sizing

NOTE 1—The Coefficient of Variation (CV) value is the standard deviation divided by the mean value for each particle size.

Min. Particle Size, x̄ (μm)	Max. Particle Size, x̄ (μm)	No. of Particles Tested	Pooled CV
262	2263	230	0.72 %

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