



Standard Practice for Microscopic Characterization of Particles from In-Service Lubricants by Analytical Ferrography¹

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1. Scope

1.1 This practice covers the identification by optical microscopy of wear and contaminant particles commonly found in used lubricant and hydraulic oil samples that have been deposited on ferrograms. This practice relates to the identification of particles, but not to methods of determining particle concentration.

1.2 This practice interfaces with but generally excludes particles generated in the absence of lubrication, such as may be generated by erosion, impaction, gouging, or polishing.

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

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D7684 Guide for Microscopic Characterization of Particles from In-Service Lubricants

G40 Terminology Relating to Wear and Erosion

¹ This practice is under the jurisdiction of ASTM Committee **D02** on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee **D02.96.06** on Practices and Techniques for Prediction and Determination of Microscopic Wear and Wear-related Properties.

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² 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.

3. Terminology

3.1 Definitions:

3.1.1 *abrasion, n*—wear by displacement of material caused by hard particles or hard protuberances. **D4175**

3.1.2 *abrasive wear, n*—wear due to hard particles or hard protuberances forced against and moving along a solid surface. **G40**

3.1.3 *adhesive wear, n*—wear due to localized bonding between contacting solid surfaces leading to material transfer between the two surfaces or loss from either surface. **G40**

3.1.4 *break-in, n*—See *run-in*. **D4175, G40**

3.1.5 *break in, v*—See *run in*. **G40**

3.1.6 *catastrophic wear, n*—rapidly occurring or accelerating surface damage, deterioration, or change of shape caused by wear to such a degree that the service life of a part is appreciably shortened or its function is destroyed. **G40**

3.1.7 *corrosion, n*—chemical or electrochemical reaction between a material, usually a metal surface, and its environment that can produce a deterioration of the material and its properties. **D4175**

3.1.8 *corrosive wear, n*—wear in which chemical or electrochemical reaction with the environment is significant. **G40**

3.1.9 *debris, n—in tribology*, particles that have become detached in a wear or erosion process. **G40**

3.1.10 *debris, n—in internal combustion engines*, solid contaminant materials unintentionally introduced in to the engine or resulting from wear. **D4175**

3.1.11 *fatigue wear, n*—wear of a solid surface caused by fracture arising from material fatigue. **G40**

3.1.12 *fretting, n—in tribology*, small amplitude oscillatory motion, usually tangential, between two solid surfaces in contact.

3.1.12.1 *Discussion*—Here the term *fretting* refers only to the nature of the motion without reference to the wear, corrosion, or other damage that may ensue. The term *fretting* is often used to denote fretting corrosion and other forms of *fretting wear*. Usage in this sense is discouraged due to the ambiguity that may arise. **G40**

3.1.13 *fretting wear*, *n*—wear arising as a result of fretting. (See *fretting*.) **G40**

3.1.14 *friction*, *n*—resistance to sliding exhibited by two surfaces in contact with each other. Basically there are two frictional properties exhibited by any surface; static friction and kinetic friction. **D4175**

3.1.15 *impact wear*, *n*—wear due to collisions between two solid bodies where some component of the motion is perpendicular to the tangential plane of contact. **G40**

3.1.16 *lubricant*, *n*—any material interposed between two surfaces that reduces the friction or wear between them. **D4175**

3.1.17 *lubricating oil*, *n*—liquid lubricant, usually comprising several ingredients, including a major portion of base oil and minor portions of various additives. **D4175**

3.1.18 *pitting*, *n*—*in tribology*, form of wear characterized by the presence of surface cavities the formation of which is attributed to processes such as fatigue, local adhesion, or cavitation. **G40**

3.1.19 *rolling*, *v*—*in tribology*, motion in a direction parallel to the plane of a revolute body (ball, cylinder, wheel, and so forth) on a surface without relative slip between the surfaces in all or part of the contact area. **G40**

3.1.20 *rolling contact fatigue*, *n*—damage process in a triboelement subjected to repeated rolling contact loads, involving the initiation and propagation of fatigue cracks in or under the contact surface, eventually culminating in surface pits or spalls. **G40**

3.1.21 *run-in*, *n*—*in tribology*, initial transition process occurring in newly established wearing contacts, often accompanied by transients in coefficient of friction, or wear rate, or both, which are uncharacteristic of the given tribological system's long term behavior. (Synonym: *break-in*, *wear-in*.) **D4175, G40**

3.1.22 *run in*, *v*—*in tribology*, to apply a specified set of initial operating conditions to a tribological system to improve its long term frictional or wear behavior, or both. (Synonym: *break in*, *v*, and *wear in*, *v*.) See also *run-in*, *n*) **G40**

3.1.23 *rust*, *n*—*of ferrous alloys*, a corrosion product consisting primarily of hydrated iron oxides. **D4175**

3.1.24 *scoring*, *n*—*in tribology*, severe form of wear characterized by the formation of extensive grooves and scratches in the direction of sliding. **D4175, G40**

3.1.25 *sliding wear*, *n*—wear due to the relative motion in the tangential plane of contact between two solid bodies. **G40**

3.1.26 *soot*, *n*—*in internal combustion*, engines, sub-micron size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion. **D4175**

3.1.27 *spalling*, *n*—*in tribology*, the separation of macroscopic particles from a surface in the form of flakes or chips, usually associated with rolling element bearings and gear teeth, but also resulting from impact events. **G40**

3.1.28 *three-body abrasive wear*, *n*—form of abrasive wear in which wear is produced by loose particles introduced or generated between the contacting surfaces.

3.1.28.1 *Discussion*—In tribology, loose particles are considered to be a “third body.” **G40**

3.1.29 *triboelement*, *n*—one of two or more solid bodies that comprise a sliding, rolling, or abrasive contact, or a body subjected to impingement or cavitation. (Each triboelement contains one or more tribosurfaces.)

3.1.29.1 *Discussion*—Contacting triboelements may be in direct contact or may be separated by an intervening lubricant, oxide, or other film that affects tribological interactions between them. **G40**

3.1.30 *two-body abrasive wear*, *n*—form of abrasive wear in which the hard particles or protuberances which produce the wear of one body are fixed on the surface of the opposing body. **G40**

3.1.31 *viscosity*, *n*—ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the pascal second (Pa.s). The centipoise (cP) is one millipascal second (mPa.s) and is often used. **D4175**

3.1.32 *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. **G40, D4175**

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *abrasive wear particles*, *n*—long wire-like particles in the form of loops or spirals generated due to hard, abrasive particles present between wearing surfaces of unequal hardness.

3.2.1.1 *Discussion*—Sometimes called cutting wear particles.

3.2.2 *analytical ferrography*, *n*—technique whereby particles from an oil sample deposited by a ferrograph are identified to aid in establishing wear mode inside an oil-wetted path of a machine.

3.2.3 *bichromatic microscope*, *n*—optical microscope equipped with illumination sources both above and below the microscope stage such that objects may be viewed either with reflected light, or with transmitted light, or with both simultaneously.

3.2.4 *black oxides of iron*, *n*—generally small, black clusters with pebbled surfaces showing small dots of blue and orange color. These are nonstoichiometric compounds containing a mixture of Fe₃O₄, Fe₂O₃ and FeO.

3.2.5 *contaminant particles*, *n*—particles introduced from an extraneous source into the lubricant of a machine or engine.

3.2.6 *chunks*, *n*—free metal particles >5 μm with a shape factor (major dimension to thickness ratio) of <5:1.

3.2.7 *corrosive wear debris*, *n*—extremely fine partially oxidized particles caused by corrosive attack.

3.2.8 *dark metallo-oxide particles*, *n*—partially oxidized ferrous wear particles indicating high heat during generation most likely due to lubricant starvation.

3.2.9 *entry*, *n*—entry area of the ferrogram, region where the sample first touches down onto the glass surface of the ferrogram and where the largest ferrous particles are found.

3.2.10 *ferrograph*, *n*—apparatus to magnetically separate and deposit wear and contaminant particles onto a specially prepared glass microscope slide.

3.2.11 *ferrogram*, *n*—specially prepared glass microscope slide that has ferrographically deposited particles on its surface.

3.2.12 *fibers*, *n*—long, thin, nonmetallic particles.

3.2.13 *friction polymers*, *n*—these are characterized by small metal particles embedded in an amorphous matrix.

3.2.14 *nonferrous metal particles*, *n*—free metal particles composed of any metal except iron. All common nonferrous metals behave nonmagnetically except nickel.

3.2.15 *nonmetallic particles*, *n*—particles comprised of compounds, organic material, glasses, etc., that have bound electrons in their atomic structure.

3.2.16 *nonmetallic amorphous particles*, *n*—particles without long range atomic order that are transparent and that do not appear bright in polarized light.

3.2.17 *nonmetallic crystalline particles*, *n*—particles with long range atomic structure that appear bright in polarized light. These may be single crystals but are most likely polycrystalline or polycrystalline agglomerates.

3.2.18 *platelets*, *n*—flat, free metal wear particles that are longer and wider than they are thick. They have a major dimension-to-thickness ratio in the range of approximately 5:1 to 10:1 or more.

3.2.19 *red oxide particles*, *n*—rust particles present as polycrystalline agglomerates of Fe_2O_3 appearing orange in reflected white light. These are usually due to water in the lubricating system.

3.2.20 *red oxide sliding particles*, *n*—sliding wear particles that appear gray in reflected white light, but are dull reddish-brown in white transmitted light.

3.2.21 *reworked particles*, *n*—large, very thin, free metal particles often in the range of 20 to 50 μm in major dimension with the frequent occurrence of holes consistent with the explanation these are formed by the passage of a wear particle through a rolling contact.

3.2.22 *rolling contact fatigue particles*, *n*—flat platelets, with their length more or less equal to their width, with smooth surfaces, random, jagged and irregularly shaped circumferences and a major dimension-to-thickness ratio in the range of approximately 5:1 to 10:1 or more.

3.2.23 *rubbing wear particles*, *n*—particles generated as a result of sliding wear in a machine, sometimes called mild adhesive wear. Rubbing wear particles are free metal platelets with smooth surfaces, from approximately 0.5 to 15 μm in major dimension and with major dimension-to-thickness ratios from about 10:1 for larger particles and to about 3:1 for smaller particles. Any free metal particle $<5 \mu\text{m}$ is classified as a rubbing wear particle regardless of shape factor unless it is a sphere.

3.2.24 *severe sliding wear particles*, *n*—severe wear particles displaying surface striations and straight edges.

3.2.25 *severe wear particles*, *n*—free metal particles $>15 \mu\text{m}$, and with major dimension-to-thickness ratios between 5:1 and 30:1.

3.2.26 *spheres*, *n*—metal spheres may be the result of incipient rolling contact fatigue or they may be contaminant particles from welding, grinding, coal burning and steel manufacturing. Spheres may also be caused by electro-pitting.

3.2.27 *wear particles*, *n*—particles generated from a wearing surface of a machine.

4. Summary of Practice

4.1 Periodic in-service lubricant samples are collected from a machine or engine as part of a routine condition monitoring program. A ferrogram is prepared from the sample to separate particles from sample fluid. The ferrogram is subsequently examined using an optical microscope to identify the types of particles present to aid in identifying the wear mode occurring in the oil-wetted path of the machine.

4.2 In usual practice of a routine condition monitoring program, a ferrogram is not prepared for every sample taken, but may be prepared when routine tests such as spectrochemical analysis, particle counting or ferrous debris monitoring indicate abnormal results.

4.3 The user of this practice employs consistent terminology to achieve accepted and understandable interpretations when communicating instructions and findings based on ferrographic analysis.

5. Significance and Use

5.1 The objective of ferrography is to diagnose the operational condition of the machine sampled based on the quantity and type of particles observed in the oil. After break-in, normally running machines exhibit consistent particle concentration and particle types from sample to sample. An increase in particle concentration, accompanied by an increase in size and severity of particle types is indicative of initiation of a fault. This practice describes commonly found particles in in-service lubricants, but does not address methodology for quantification of particle concentration.

5.2 This practice is provided to promote improved and expanded use of ferrographic analysis with in-service lubricant analysis. It helps overcome some perceived complexity and resulting intimidation that effectively limits ferrographic analysis to the hands of a specialized and very limited number of practitioners. Standardized terminology and common reporting formats provide consistent interpretation and general understanding.

5.3 Without particulate debris analysis, in-service lubricant analysis results often fall short of concluding likely root cause or potential severity from analytical results because of missing information about the possible identification or extent of damaging mechanisms.

5.4 Ferrographic analysis, as described in this practice, provides additional particle identification capabilities beyond methods described in Guide D7684 for the following reasons:

(1) The ferrographic particle separation method is magnetic thus making it possible to readily distinguish between ferrous and nonferrous wear particles.

(2) Ferrography separates ferrous (magnetic) particles by size.

(3) Deposition is on a glass substrate so that particles may be examined using transmitted light as well as reflected light allowing particle types to be identified that cannot be identified when examination is done using only reflected light.

(4) Ferrograms may be heat treated providing important distinctions between ferrous alloy types (steel versus cast iron), further distinctions among various nonferrous alloys and distinctions between inorganic and organic particles.

5.5 Caution must be exercised when drawing conclusions from the particles found in a particular sample, especially if the sample being examined is the first from that type of machine. Some machines, during normal operation, generate wear particles that would be considered highly abnormal in other machines. For example, many gear boxes generate severe wear particles throughout their expected service life, whereas just a few severe wear particles from an aircraft gas turbine oil sample may be highly abnormal. Sound diagnostics require that a baseline, or typical wear particle signature, be established for each machine type under surveillance.

6. Apparatus

6.1 Required Components:

6.1.1 *Ferrograph or Ferrogram Maker*—Apparatus for magnetically separating particles from fluids.

6.1.2 *Bichromatic Microscope*—An optical microscope is required with dry metallurgical objective lenses and equipped with a reflected light source and a transmitted light source so that objects may be viewed from both above and below the microscope stage. This permits objects to be viewed either with reflected light, or with transmitted light, or with both simultaneously. Bichromatic microscopes for ferrogram examination are required to be equipped with three objective lenses to give varying degrees of magnification. The low magnification objective lens is typically 10×, the medium magnification objective lens may be 40× or 50× and the high magnification objective lens may be 80× or 100×. Ten power (10×) ocular (eyepiece) lenses are used such that total magnification achieved is 100× at low magnification, 400× or 500× at medium magnification and 800× or 1000× at high magnification. The numerical apertures of the objective lenses need to be high to maximize illumination of particle surfaces when viewed in reflected light. It is required to be able to polarize either light path to facilitate particle identification. Polarized light aids in the identification of nonmetallic particles. A red filter is required to be optionally placed in the reflected light path and a green filter is required to be optionally placed in the transmitted light path. The simultaneous use of red reflected and green transmitted light aids in the distinction between metallic and nonmetallic particles. One of the ocular lenses should be fitted with a calibrated scale so that length of objects may be measured. The stage drive of the microscope should be fitted with calibrated divisions so that thickness of objects may be measured as the stage is raised or lowered.

6.1.3 *Blank Ferrogram Glass Substrates*—A supply of specially prepared microscope slides with nonwetting barriers to contain sample flow in the central portion of the substrate are required.

6.1.4 *Precision Pipettor*—A pipettor capable of delivering a precise volume of 1 mL of viscous fluid is required.

6.1.5 *Mixing Vials*—Clean vials, usually 12 mL capacity, are needed to mix sample with solvent prior to processing by the ferrograph.

6.2 Optional Components:

6.2.1 *Hot Plate*—A hot plate capable of achieving surface temperatures of 540°C is required if it is desired to heat treat ferrograms to further identify the metallurgy of metal particles.

6.2.2 *Surface Thermometer*—A surface thermometer capable of measuring to 540°C is needed for heat treating ferrograms.

6.2.3 *Tongs*—Tongs are needed to remove the heated ferrogram from the hot plate.

6.2.4 *Camera*—The microscope may be equipped with a suitable camera for taking photomicrographs for reporting and documenting purposes.

7. Reagents

7.1 *Heptane*—The recommended solvent is heptane, but other solvents may be used if they meet the following criteria:

7.1.1 The solvent must be a good oil solvent.

7.1.2 The solvent cannot be too volatile. If the solvent evaporates too quickly the strings of particles on the ferrogram surface will be pulled out of place by the movement of the quickly drying solvent.

7.1.3 If the solvent evaporates too slowly, excessive time will be spent waiting for the ferrogram to dry.

7.1.4 The solvent needs to be residue and particle-free. Prepare a ferrogram using only solvent and examine it under the microscope to make sure the ferrogram surface is clean. From a practical viewpoint, it will be almost impossible to prepare a blank ferrogram, that is, one that is totally free of particles. Therefore, some judgment should be exercised regarding an acceptable cleanliness level. A few small nonmetallic particles are tolerable in that they would not interfere with evaluation of machine condition. On the other hand, if the blank ferrogram has metallic particles deposited on it, then steps need to be taken to eliminate the source of contamination. It may be necessary to filter the solvent through a submicron membrane filter to remove particulate contaminants or to let the solvent remain undisturbed for overnight or longer so that particles settle to the bottom of the bottle or container. Withdrawing solvent from near the top of the undisturbed container will likely yield particle free solvent.

8. Sampling and Sample Handling

8.1 *Sample Acquisition*—The objective of sampling is to obtain a test specimen that is representative of the entire quantity. Thus, laboratory samples should be taken in accordance with instructions in Practice D4057.

9. Procedure

9.1 Ferrogram Preparation:

