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AEROSPACE STANDARD



AS7101

**REV.
A**

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NATIONAL AEROSPACE AND DEFENSE CONTRACTORS ACCREDITATION PROGRAM (NADCAP) GENERAL REQUIREMENTS FOR MATERIALS TESTING LABORATORY ACCREDITATION PROGRAM

1. SCOPE:

1.1 This standard establishes the minimum requirements for Materials Testing Laboratories (MTL) accredited by NADCAP. The following test methods and processes for metallic material systems are covered in this standard.

- a. Chemical Testing
- b. Mechanical Testing
- c. Metallography and Microhardness
- d. Hardness
- e. Corrosion
- f. Mechanical Testing Specimen Preparation
- g. Differential Thermal Analysis
- h. Heat Treating
- i. X-Ray Diffraction
- j. Fastener Testing

2. REFERENCES:

2.1 Applicable Documents

2.1.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AS7001 National Aerospace and Defense Contractors Accreditation Program (NADCAP) - Program Description

AS7002 National Aerospace and Defense Contractors Accreditation Program (NADCAP) - Rules for Implementation

AS7003 National Aerospace and Defense Contractors Accreditation Program (NADCAP) - Program Operation

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2.1.1 Continued

AMS 2248	Chemical Check Analysis Limits - Wrought Corrosion and Heat Resistant Steels and Alloys, Maraging and Other Highly-Alloyed Steels
AMS 2249	Chemical Check Analysis Limits - Titanium and Titanium Alloys
AMS 2259	Chemical Check Analysis Limits - Wrought Low-Alloy and Carbon Steels
AMS 2268	Chemical Check Analysis Limits - Cast Nickel and Nickel Alloys
AMS 2269	Chemical Check Analysis Limits - Wrought Nickel and Nickel Alloys
AMS 2280	Trace Element Control, Nickel Alloy Castings
AMS 2750	Pyrometry
SAE J784	Residual Stress Measurement by X-Ray Diffraction
SP 453	Retained Austenite and its Measurements by X-Ray Diffraction

2.1.2 PRI Publications: Available from PRI, 161 Thornhill Road, Warrendale, PA 15086-7527.

PRI AC7101/1A	General Requirements
PRI AC7101/2A	Chemical Analysis
PRI AC7101/3A	Mechanical Testing
PRI AC7101/4A	Metallography & Microhardness
PRI AC7101/5A	Hardness
PRI AC7101/6A	Corrosion
PRI AC7101/7A	Mechanical Test Specimen Preparation
PRI AC7101/8A	Differential Thermal Analysis
PRI AC7101/9A	Heat Treating
PRI AC7101/10	X-Ray Diffraction Evaluation
PRI AC7101/11	Fastener Testing
PRI AC7108	Chemical Processing
PRI AC7109/5	Coating Evaluation Laboratory Practices

2.1.3 ASTM Publications: Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428

ASTM B 117	Method of Salt Spray (Fog) Testing
ASTM B 214	Test Method for Sieve Analysis of Granular Metal Powders
ASTM E 3	Methods of Preparation of Metallographic Specimens
ASTM E 4	Standard Practices for Load Verification of Testing Machines
ASTM E 8	Test Methods of Tension Testing of Metallic Materials
ASTM E 10	Test Method for Brinell Hardness of Metallic Materials
ASTM E 18	Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
ASTM E 21	Test Methods for Elevated Temperature Tension Tests of Metallic Materials
ASTM E 23	Methods for Notched Bar Impact Testing of Metallic Materials
ASTM E 29	Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications
ASTM E 45	Practice for Determining the Inclusion Content of Steel

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ASTM E 74	Standard Practice of Calibration of Force-Measuring Instruments for Verifying the Load In Direction of Testing
ASTM E 82	Method for Determining the Orientation of a Metal Crystal
ASTM E 83	Standard Practice for Verification and Classification of Extensometers
ASTM E 92	Test Method for Vickers Hardness of Metallic Materials
ASTM E 112	Methods for Determining Average Grain Size
ASTM E 139	Practice for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials
ASTM E 140	Hardness Conversion Tables for Metals
ASTM E 220	Method for Calibration of Thermocouples by Comparison Techniques
ASTM E 228	Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer
ASTM E 290	Test Method for Semi-Guided Bend Test for Ductility of Metallic Materials
ASTM E 292	Practice for Conducting Time for Rupture Notch Tension Test of Materials
ASTM E 340	Method for Macroetching Metals and Alloys
ASTM E 384	Test Method for Microhardness of Materials
ASTM E 399	Test Method for Plane-Strain Fracture Toughness of Metallic Materials
ASTM E 407	Methods for Microetching Metals and Alloys
ASTM E 466	Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials
ASTM E 606	Practice for Strain-Controlled Fatigue Testing
ASTM E 647	Test Method for Measurements of Fatigue Crack Growth
ASTM E 743	Standard Guide For Spectrochemical Laboratory Quality Analysis
ASTM E 851	Standard Practice For Evaluation Of Spectrochemical Laboratories
ASTM E 876	Practice for Use of Statistics in the Evaluation of Spectrometric Data
ASTM E 915	Method of Verifying the Alignment of X-Ray Diffraction Instrumentation of Residual Stress Measurement
ASTM E 930	Methods of Estimating the Largest Grain Observed in a Metallographic Section
ASTM E 975	Practice for X-Ray Determination of Retained Austenite In Steel with Near Random Crystallographic Orientation
ASTM E 1012	Practice for Verification of Specimen Alignment Under Tensile Loading
ASTM Volume 3.01	Metals - Mechanical Testing: Elevated and Low-Temperature Tests; Metallography
ASTM Volume 3.02	Wear and Erosion; Metal Corrosion
ASTM Volume 3.05	Chemical Analysis of Metals and Metal Bearing Ores

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- 2.1.4 Military Publications: Available from Naval Publications and Forms Center, Attn: NPODS, 5801 Tabor Avenue, Philadelphia, PA 19120-5099.

MIL-H-6875 Process for Heat Treatment of Steel
ISO 10012-1 Quality Assurance Requirements for Measuring Equipment
ANSI Z540-1 General Requirements for Calibration Laboratories and Measuring & Testing Equipment

2.2 Definitions

ACCEPTANCE TESTS/PROPERTIES: Tests/properties which are required to be included on each certification for acceptance of the delivered product (for example: Tensile, stress rupture, hardness, and metallographic tests).

CAPTIVE LABORATORY: A laboratory that belongs to a material supplier, with systems that are dependent on those of the supplier, and with testing capabilities that are limited to those required by the supplier's material.

CERTIFICATE OF CONFORMANCE: Certificate issued by the laboratory which documents conformance of material to the material specification and which may describe the testing performed, but does not include the numerical values of results obtained.

CERTIFICATE OF TEST, TEST REPORT: Document issued by the laboratory describing the testing performed, the specific results obtained, and whether results conform to the material specification.

CHECK ANALYSIS: An analysis made by the purchaser or supplier of the metal after it has been worked into semi-finished or finished forms or fabricated into parts. This analysis is either for the purpose of verifying the composition of a heat or lot or to determine variations in the composition within the product on the basis of this check analysis. In the analysis of finished parts, these values do not apply to elements whose percentage can be varied by fabricating techniques employed (e.g., surface hydrogen in steels and titanium alloys) unless the sample is taken in such a manner as to exclude such changes. [This definition taken from AMS Check Analysis specifications.]

CUSTOMER: The company who places the order for testing with the laboratory.

INDEPENDENT LABORATORY: A laboratory whose systems are not dependent on those of specific material suppliers. (Ownership by a material supplier does not exclude a laboratory from being considered 'Independent'.)

INSTRUMENTAL ANALYSIS: Includes SELF-CALIBRATING TESTS, and other testing which is essentially controlled by the equipment but which may not be independent of operator technique and/or interpretation. Restrictions, requirements, and examples are given in the sections for test families.

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INVALID TEST VALUE: A test value considered to be untrue because it does not fit the population of other values from the same sample and the non-validity is evaluated and reported per Appendix B.

LOW CYCLE FATIGUE (LCF): Fatigue test characterized by hysteresis behavior from loading-unloading in plastic region. Generally strain control per ASTM E 606, but may include load-control and torsional LCF testing. Note: Hysteresis behavior may only be exhibited during the first full cycle.

LOW STRESS GRINDING (LSG): Grinding/polishing under controlled conditions to minimize and produce required compressive surface stresses.

MATERIAL CERTIFICATION: The certificate of Test or Conformance issued by the laboratory.

MATERIAL SPECIFICATION: The document(s) describing which tests are to be performed and the conformance limits required/expected, and (sometimes) the test specification(s) to be used. This definition can include drawings or other purchasing documents.

MATERIAL SUPPLIER: A company who supplies material under the Purchaser's order (e.g., under Purchase Order or cooperative agreements).

NIST: National Institute of Standards and Technology.

NON-CONFORMING TEST RESULT: A test result that does not conform to the material specification.

PROCUREMENT DOCUMENT: The Purchase Order (P.O.) that establishes contractual quality requirements for the material. In certain business arrangements which do not include issuance of P.O.s (e.g., revenue share agreements) imposition of this specification is by way of the Quality Plan.

PURCHASER: The procuring activity that issued the procurement document, for material or services, which invoked this document.

QUALITY SYSTEM DOCUMENT: A manual prepared by the laboratory, that contains the guidelines for establishment and maintenance of Quality-related activities and procedures, which is approved by the Quality function and company management.

RE-TEST: A repeat of a TEST by the same laboratory, using the same method, equipment (of equivalent accuracy or better), and sample. Usually performed in response to suspect or non-conforming results from the original test(s).

REFEREE TEST: A repeat of a TEST from the same SAMPLE by a different laboratory, or using more accurate or precise methods/equipment.

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REPLACED TEST ("NO-TEST"): A test whose results are considered to be untrue because of identified causes other than properties of the material being tested (e.g., errors in specimen machining or testing).

REPLACEMENT TEST: A test made as a result of a Replaced Test.

RESIDUAL ELEMENT: An unspecified element, originating in raw materials, melting equipment (e.g., furnace refractory lining), or melting fluxes-slugs-atmospheres defined by the melting practice, or as defined by specification.

ROUND ROBIN TESTING: Testing of specimens from the same sample by different laboratories and/or by different test methods or equipment. Requirements and restrictions are given in the sections for the generic test types.

SAMPLE: Material supplied for testing. (see also "SPECIMEN").

SELF-CALIBRATING TEST METHOD: Testing that is essentially controlled by and results reported by the equipment, independent of operator technique (other than specimen loading), for which equipment is calibrated by testing certified standard specimens prior to and after testing required specimens. Examples, requirements, and restrictions are given in the sections for the generic test types.

SIGNIFICANT OUT OF TOLERANCE CONDITION: A condition in the laboratory that results in the change of the disposition of material (e.g., from conforming to nonconforming).

SPECIMEN: A portion of the **SAMPLE** selected for testing to accurately represent the entire **SAMPLE**.

SQA: Software Quality Assurance. A program to ensure software used to control tests and/or generate data is not altered without adequate validation and documentation control.

SUBSTANTIATION: All of the following terms mean the same thing.

- Substantiation
- Source Substantiation (SS, S.S., or S/S)
- VSE (Vendor Substantiation Engineering)

SUBSTANTIATION TEST: Any test whose result is required for Substantiation. These include (but are not limited to) all Acceptance and Capability tests established by drawing.

SUB-TIER LABORATORY: A laboratory which does not belong to a direct material supplier. Systems for such laboratories must qualify as "INDEPENDENT".

TEST CODE: The single-letter or double-letter code denoting the specific test type qualified.

NOTE: Double-letter codes will have "X" prefixes.

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TEST RECORDS: Records maintained by the laboratory; to be available for review, but not required to be furnished with the certification unless specified.

TEST SPECIFICATION: Document describing the method(s) and procedures(s) by which material is to be tested.

TRACE ELEMENT: A residual element that occurs in very low concentrations, generally less than 0.01%.

WORKING STANDARD: Standardized material used during routine testing to ensure that testing equipment is properly calibrated for the material which requires testing.

3. SURVEY REQUIREMENTS:

3.1 Survey information has been divided into parts as follows:

PRI AC7101/1 GENERAL REQUIREMENTS, ALL LABORATORIES

This section contains general information regarding quality systems that would be collected for a survey of any laboratory.

PRI AC7101/2A CHEMICAL TESTING

PRI AC7101/3A MECHANICAL TESTING

PRI AC7101/4A METALLOGRAPHY & MICROHARDNESS

PRI AC7101/5A HARDNESS

PRI AC7101/6A CORROSION

PRI AC7101/7A MECHANICAL TESTING SPECIMEN PREPARATION

PRI AC7101/8A DIFFERENTIAL THERMAL ANALYSIS

PRI AC7101/9A HEAT TREATING

PRI AC7101/10 X-RAY DIFFRACTION

PRI AC7101/11 FASTENER TESTING

3.1.1 These PRI Audit Criteria form a part of this standard to the extent referenced herein.

This information package will be reviewed by NADCAP and approval to schedule the audit will be obtained. PRI will coordinate with the laboratory to obtain any additional information in support of the audit.

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4. REQUIREMENTS: ALL LABORATORIES:

4.1 Laboratory Accreditation

Initial laboratory accreditation is for a three year period. After this initial accreditation, an annual Reduced Scope Audit must be successfully completed to continue the accreditation term of qualifying laboratories. If a laboratory does not qualify for a Reduced Scope Audit, a complete re-audit must be successfully completed yearly to continue the accreditation term for the laboratory. Accreditation requires all of the following:

- a. Conformance to applicable provisions of this standard.
- b. Completion by the laboratory of matrices describing facilities and calibration practices.
- c. Acceptable evaluation to NADCAP checklists for the applicable codes which are derived from this standard, from standard testing specifications (e.g., ASTM), and from recognized standards of good laboratory practice.

4.1.1 Checklists for each test type are designed to utilize "YES/NO" questions. All "No" answers require an explanation. A "not applicable" explanation is not acceptable without further information.

Corrective action is required for any NADCAP Standard violation.

4.2 Quality Organization

The Quality Assurance function shall report directly to the highest level of management within the laboratory, and this shall be documented via a company flow chart. Quality Assurance shall be operationally independent from other functions of the laboratory, and shall have "stop work" authority ("shipment hold" for captive laboratories) to the degree necessary to ensure that data released conforms to the applicable Quality requirements. The laboratory shall be organized in such a way that confidence in its independence of judgement and integrity is maintained at all times.

The Q.A. function shall participate in the organization and planning of the following:

- a. Definition and selection of equipment
- b. Maintenance and calibration of facilities and equipment
- c. Test procedures (revision and implementation)
- d. Training and qualification of personnel
- e. Selection and survey of sub-contractors

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4.2.1 Quality System Document (QSD)

Each laboratory is required to have a Quality System Document, which is approved by the Q.A. authority and laboratory management. The Quality System Document shall comply with either a national or international standard. Captive laboratories may be covered by the quality manual of the material supplier plus detailed manuals covering laboratory operations. The manual and documents referenced therein shall cover all requirements of this standard including approval of non-traditional and alternate procedures permitted by this standard. Changes shall be approved by the Q.A. authority and recorded on a revision page; any changes which might affect conformance to this standard require a notification to NADCAP for evaluation of accreditation status. Changes shall be made available for the NADCAP audit. Distribution of the manual shall be controlled to assure currency of the revisions used within the laboratory.

4.3 Facility

The following factors will be considered in NADCAP accreditation of the laboratory, and may be cause for accreditation to be refused.

- a. Testing environment free from external vibrations, electrical transients, voltage fluctuations, RF pollution, and atmospheric contamination. These factors shall have been considered in design of the facility itself or of individual equipment sets.
- b. Temperature and humidity control. (See specific tests.)
- c. Cleanliness, housekeeping, and orderly handling of material.
- d. Facility must have a basic safety program.
- e. Sensitive electrical equipment shall not be subjected to corrosive environments (e.g., analytical computers are not to be located in the sample dissolution area).

4.4 Systems

4.4.1 Laboratory systems shall include provisions for self-audit. A routine, internal audit program shall be described in writing and carried out according to a written format and to a published schedule.

Internal audits are carried out to check:

- a. Correct application of testing methods
- b. Compliance with customer orders
- c. Correct application of procedures
- d. Correct preparation of certificate
- e. Cause and corrective actions for revealed discrepancies

Internal audit reports shall be transmitted to the laboratory management with the application of corrective action controlled. The Q.A. function shall:

- a. Use a central log to record deviations and corrective actions
- b. Investigate to determine probable cause
- c. Participate in the development of corrective actions
- d. Control the application of corrective actions

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4.4.2 The laboratory shall maintain a system which ensures access to current issues of applicable specifications.

4.5 Personnel

A central file shall be maintained convenient to the laboratory, containing the names and relevant qualifications (education + training + experience) of laboratory personnel responsible for test results reported on certifications. If degreed personnel "or equivalent" are required, justification for "equivalent" shall be documented. The Quality System Documents shall define equivalency requirements. Records shall identify key personnel (i.e., personnel whose knowledge or techniques are essential to proper performance of the test); records shall be made available for audit. Notification of NADCAP is required for changes in specific key personnel.

4.6 Procedure System

The laboratory shall maintain controlled, written procedures for the following:

- a. Issue and control of quality-related documents
- b. Sub-contracting activities
- c. Procurement activities
- d. Calibration functions
- e. Records of calibration results
- f. Records of job orders and follow-up of samples
- g. Detailed testing methods (specific for the laboratory)
- h. Transmission of results
- i. Records of results
- j. Record retention
- k. Validation of data analysis and presentation software
- l. Disposition of samples (e.g., retain, return to customer)

4.6.1 Test Procedures

Written procedures or computer programs, under revision control and (as applicable) Software Quality Assurance control, shall be issued and followed for all applicable tests. Procedures shall reference testing specifications from which they are derived, and shall include specimen location and preparation. They shall be sufficiently detailed so that the test can be consistently reproduced in that laboratory. General procedures (e.g., ASTM E 8) which contain two or more optional methods for individual operations are not satisfactory for this purpose. Each procedure shall evidence the individual/organization responsible for issue and control. A procedure shall ensure that changes to a testing method are not applied without authorization, in writing, and provide for removal of obsolete test methods.

4.6.2 Non-Standard Test Procedures: Work completed in support of special investigations shall be fully documented using test logs per paragraph 4.12 and test reports per paragraph 4.13. All procedures used during these non-standard tests shall be documented to the extent that the test could be reproduced.

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4.6.3 The laboratory shall have a procedure which ensures that the information required by Appendix A of this standard, as applicable, is received prior to commencing work.

4.7 Specimen Identification and Tracking

A unique set of identifying numbers shall be assigned to each test to ensure traceability from the certificate issued to the customer order, material tested, test methods used, test results and original cutting plan. The laboratory's systems shall provide for tracking and accountability of all specimens. Specimens shall be stamped, tagged or otherwise identified to avoid inter-mixing, and shall remain identifiable until disposal.

4.7.1 Specimen Handling

A procedure shall define specimen handling policies which include internal processing, shipping of specimens and specimen protection for possible future examination. Provisions for handling in accordance with contract requirements shall be included in the procedure. Shipping documents shall be prepared to accompany specimens, and shall include sufficient traceability to correlate the document with the specimens. The shipping document shall be checked prior to issuance.

4.8 Equipment Calibration and Maintenance

The laboratory shall establish a calibration schedule for each piece of equipment. This schedule shall include frequencies, recalls and responsibility for calibration (source). This section is applicable to both internal and external calibrations. All calibrations shall be documented by calibration certificates. The certificates shall contain the following information, as a minimum:

- a. Description of equipment and operating range, including the precision of the instrument being calibrated
- b. Agency/individual performing the calibration
- c. Traceability of primary standards and their accuracy
- d. Identification of the calibration procedure used
- e. Date of calibration and recall
- f. The as found condition of the instrument
- g. Signature of authorized calibration personnel
- h. Environmental conditions at time of calibration

Written procedures, referenced in the Quality System Documents, shall be provided and utilized for calibration of all measuring and testing equipment and measurement standards which are pertinent to certified results (ref. ISO 10012-1, ANSI Z540-1). Calibrations shall be traceable to NIST or other sources as described in ISO 10012-1 or ANSI Z540-1. If an outside agency performs calibration, they shall be required to provide such procedures. As a minimum, the procedures shall specify the precision of the instrument being calibrated, the accuracy of the standards used and their traceability to primary standards, the minimum frequency of calibration, and detailed calibration

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4.8 Continued

instructions (e.g., for laboratory furnace calibration, the thermocouple type and placement, and rules for temperature stabilization). Calibration shall be summarized for each laboratory using a matrix located in the applicable checklist. All equipment not under calibration control shall be explained and so identified. New or repaired equipment shall be calibrated prior to being released for testing purposes. All calibrated equipment shall be handled and stored in a manner that prevents damage. A procedure shall define the method used to protect pre-set equipment from tampering. Computer-controlled equipment shall be calibrated as a system.

4.8.1 Preventive maintenance shall be in accordance with a written plan, and shall be logged. There shall be evidence that this plan has been implemented. The plan shall discuss any major equipment. Calibration may be considered part of the laboratory's plan. Preventive maintenance is not required if Statistical Process Control indicates no deterioration in equipment performance. Equipment shall be maintained in such a condition so as not to introduce testing error.

4.8.2 Calibration and maintenance records shall be available upon request.

4.8.3 Calibration Identification. Calibration status stickers (last/next) should be posted on each test machine. "Calibration Not Required" or "For Reference Only" stickers should be used where applicable.

4.8.4 Standards shall be clearly identified to prevent mixing between calibrating (primary), and calibrated (working) and non-calibrated equipment and standards.

4.8.5 Out of service equipment shall be identified and segregated from equipment in use.

4.8.6 A procedure shall address the calibration and usage of "employee-owned" equipment used for acceptance purposes.

4.8.7 The Q.A. function shall document the review and acceptance of calibration certificates, and ensure that the procedures are followed.

4.9 Replacement Testing and Re-Testing

A written policy for replacement testing and re-testing in accordance with customer requirements shall be established and referenced through the Quality System Document.

4.9.1 Accountability: All specimens provided to the laboratory shall be accounted for on the Certificate or (for Captive Laboratories) the test report, whether tested to completion or failure, or replaced. When sufficient material is available for replacement specimens to be made by the laboratory from the same sample, replaced specimens need not be reported on the Certificate, but should be documented in the laboratory's internal records.

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4.9.2 Invalidation of test results is permitted only when authorized by the material specification, or other contractual documents. Invalid test results shall be evaluated and reported per Appendix B.

4.9.3 The laboratory's procedures and training shall provide for recognition of invalid test data.

4.10 Errors in Testing

A written policy shall be established and referenced through the Quality System Documents which includes designation of individuals (or job titles) responsible for action and notification (if required). When errors in testing are detected which could result in a significant out-of-tolerance condition (ISO 10012-1, ANSI Z540-1), the suspect population shall be defined and documented. If all affected material is not contained and correctness of results verified, all customers to whom the laboratory sent incorrect or suspect test results shall be notified within five (5) working days.

Example: "Significant out-of-tolerance" conditions might include excursions exceeding chemical check analysis limits, exceeding the precision claimed by capability matrices, or exceeding 25% of the tolerance band. All tests since the last correct calibration are suspect unless the population can be further restricted.

4.11 Certificate of Test/Conformance

Tests performed shall be documented and all valid results shall be reported in a test report or certification. For captive laboratories, this is controlled by a Q.A. function. The certification shall be signed and released, only after a review of the data for completeness and accuracy.

4.11.1 Contents: The following items shall be included in each Certificate, in addition to requirements established by the applicable specification:

- a. The laboratory's identification: Name and Address.
- b. The company for whom the testing was performed.
- c. The number(s) and issue(s)/revision(s) of the specification(s) against which material was tested.
- d. The testing specification used, if not called out by the material specification. Any non-standard practices must be disclosed.
- e. Certificate of Test: Include the numerical results of all tests and inspections performed for which the material specification establishes numerical requirements. When certifying to a specific specification (as opposed to all certifications which may later be referenced against many individual specifications), the certificate shall show that the results are in accordance with the requirements of that specification or the tests performed.

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4.11.1 Continued

EXAMPLE: Specification limits should be included on the certificates (either by the laboratory or material supplier), in such a manner as to facilitate comparison of material specification requirements versus test results, and to ensure that all items (e.g., all chemical elements) have been evaluated.

- f. Description of the results (e.g., conform/non-conform) of all tests for which the material specification does not establish numerical requirements. Report any unusual observations (identify as "Observation"). The separation of conforming and non-conforming results on separate certificates is not permitted.
- g. Unique identification on each certification shall distinguish the original from a copy.
- h. The certificate or report shall contain a statement that it shall not be reproduced except in full without the written approval of the laboratory, and that the recording of false, fictitious, or fraudulent statements or entries on this document may be punished as a felony under federal law.

4.11.2 Preparation of Certificate: Each page of the certificate shall be numbered "page ___ of ___", and shall have unique identification traceable to the job and laboratory identification. Use of 'white-out' or erasures on the signed certificate, test record documents or copies thereof is prohibited; corrections may be lined out and initialed. Test results for all specimens in the same set shall be included on the same certificate; that is, separate certificates shall not be issued for conforming versus nonconforming material. Re-test values, and values known to be nonconforming (i.e., when compared against mandatory specification values), shall be clearly identified as such. All specimens provided to the laboratory must be accounted for, e.g., by stating "Specimen 4A253: No Test - broke in grips." Test values from replaced specimens need not be reported.

Computer generated certificates require either (a) an actual or facsimile signature, or (b) a letter accompanying the certificate, signed by the laboratory's manager attesting that the laboratory is using a computerized system, the typed name on the document is an authorized employee, and the laboratory is responsible for the information it contains.

4.11.3 A Certificate of Conformance (in lieu of a Certificate of Test), shall not be provided unless specifically required by the material specification.

4.11.4 Statement of Conformance: When testing is performed against material specifications that require certificates to state that a test "conforms to specification", conformance shall be clearly noted; statements to the effect that "values are true and accurate" are not sufficient.

4.11.5 Appendix C lists errors most frequently found on certificates. This Appendix should be used for audit by both the laboratory and the material supplier.

4.11.6 Electronic Data Interchange (EDI) will be controlled in accordance with ANSI Standard ASC X12 (when implemented).

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- 4.11.7 When custom software is used for data compilation, it shall be subjected to Software Quality Assurance.
- 4.11.8 Revisions to certificates shall be handled in one of the following manners, or a procedure shall specify the method used by the laboratory:
- a. A new certificate number is issued, and referenced to the erroneous certification.
 - b. The same certification number, with errors identified, the certification marked as "corrected", initialed and dated.
- 4.12 Test Logs
- A numbered and bound log, or equivalent system that prevents substitution or deletion of data, shall be used.
- 4.12.1 Logging - All Tests: All tests shall be logged against the unique identifying numbers assigned for traceability, and shall be traceable to the specific testing procedure used and test data generated.
- 4.12.2 Logging - Replaced Tests, Invalidated Tests, Nonconforming Tests, and Re-Tests: Such tests shall be identified as such, and cross-indexed against each other where applicable. Reason(s) for replacement testing or re-testing shall be noted. Laboratory management authorization shall be noted for re-tests not specifically authorized by the laboratory's customer. Logs shall be reviewed at least quarterly for trends which could indicate deterioration in testing disciplines.
- 4.13 Test Records
- Records shall be maintained, traceable to each certificate issued, which would permit reproducing the test method in that facility, or permit identification of suspect tests if any element of testing were found to be incorrect at a later date. Examples of items which should be recorded are: location of specimen(s) within the sample, equipment set (test stand, tooling, recorders, etc.), temperature and dimensional tolerances (if not reported on the certificate), comparison standards, personnel.
- 4.14 Retention and Recovery
- Minimum retention times are separate from requirements that may be placed on the material supplier by the Purchaser, and apply after the certification is issued to the customer. Recovery shall require a maximum of 3 working days.
- a. Test Data (including metallographic exhibits) relevant to material certification, including Test Logs which relate test results to the material tested, supporting data (e.g., tensile test printouts), and calibration data shall be defined via the Quality System Documents, and shall be retained as specified by the customer. **NOTE:** The material supplier is required to retain test data relevant to certification of substantiated material for 30 years minimum, or as specified by the customer.

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- b. Test material (specimens and extra sample material) not returned to the laboratory's customer shall be retained for 6 months. Special requirements may be established for the retention of hazardous materials if required by local or company safety codes.
- c. The laboratory shall implement a system to provide back-up storage of certificates and/or test reports, in case of fire, water damage, or other cause which could destroy the originals.

4.14.1 The following information, as a minimum, is considered to be part of a test record, and as such, is required to be maintained and recoverable:

- a. Customer and order identification
- b. Part or sample identification
- c. Identification of tests conducted
- d. Applicable specifications
- e. Test results
- f. Copy of the certification issued to customer
- g. Copy of certifications for nonconforming results
- h. Copy of revised certifications issued
- i. Identification of subcontractors

4.15 Round Robin Testing

Each laboratory shall participate in Round Robin (R/R) program(s). While participation in external laboratory programs is desired, intra-laboratory programs may be acceptable unless otherwise specified (e.g., chemical and LCF laboratories require inter-laboratory programs). Round Robin Programs shall be administered in accordance with Appendix E of this standard. Some expected elements of R/R programs are as follows:

- a. Material supplied by one source and controlled for uniformity. Specimen sets are prepared by one or more participants, with duplicate specimens from each set tested by each participant and/or test method.
- b. (External Laboratory Program) Participating laboratories identified by code to minimize the possibility of bias during data analysis.
- c. Statistical analysis and correlation performed on results.
- d. Results used to identify and correct problems, and/or to initiate changes in testing methods or practices (e.g., action taken if laboratory's results are more than 2 sigma from mean value).
- e. Frequency of Round Robin testing is defined in Appendix E.
- f. Round Robin programs require documentation and evidence of corrective action implementation.

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4.15.1 Internal Round Robin programs shall be structured to evaluate the following:

- a. Data obtained from different machines used for the same test.
- b. The same test performed by different technicians.

4.16 Security

Security shall be provided to protect the confidentiality of customer specifications.

4.17 Audit

All procedures and records required by this standard are subject to audit at the laboratory facility.

4.18 SUB-CONTRACTING

The laboratory shall utilize qualified sources for all sub-contracted processes. The source shall be approved by the Quality function based on NADCAP accreditation, or at least three (3) of the following attributes:

- a. Documentation
- b. Documentation and initial survey
- c. 100% inspection of product/service
- d. Sampling inspection of product/service
- e. Periodic on-site survey by the Q.A. function

4.18.1 The laboratory shall have access to sub-contractor facilities for the purposes of Quality Audits.

4.18.2 Quality Assurance requirements specified in the contract shall be transmitted to the sub-contractor.

4.18.3 All qualified sub-contractors shall be listed on the laboratory's Approved Vendors List.

4.19 Training and Qualification

Definitions of technical personnel functions shall be documented, which describe special skills required for the position. Testing, data review and preparation personnel shall be qualified through the following, as applicable:

- a. Training by individual with a technical degree and/or related experience.
- b. Periodic overcheck of work (documented for testing personnel).
- c. Round Robin comparison among personnel (testing staff).

Training activities shall be documented in the Q.A. files, and technical personnel shall be listed in Figure 1B of AC7101/1A.

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4.20 Testing Personnel

All personnel shall be trained in accordance with a written plan, which shall include general and specific aspects of the procedure to be performed. Operators shall be trained to recognize proper operation of equipment.

Laboratory management shall assign responsibility for review and approval of tests, training and qualification of technicians, standards and procedures.

5. CHEMICAL TESTING

5.1 Test Types/Codes

- D Wet Chemistry (Gravimetric)
- F Atomic Emission Spectroscopy
 - F1 Direct Current Plasma (DCP)
 - F2 Inductively Coupled Plasma (ICP)
 - F3 Spark/Arc (OES)
 - F4 Glow Discharge
- G Elemental Analysis (Combustion or Fusion)
 - G1 Carbon
 - G2 Hydrogen
 - G3 Nitrogen
 - G4 Oxygen
 - G5 Sulfur
- S X-Ray Fluorescence
- V Mass Spectrometry
- W Atomic Absorption
 - W1 Flame
 - W2 Graphite Furnace

5.1.2 Alloy Families for Chemical Testing

- | | |
|---------|---------------------|
| Ni Base | Mg Base |
| Co Base | Cu Base |
| Ti Base | Fe Base, Low Alloy |
| Al Base | Fe Base, High Alloy |

5.1.3 Additional Chemistry Definitions: Definitions of and requirements for the following terms relevant to chemical analysis are given in AMS 2248, AMS 2249, AMS 2259, AMS 2268, and AMS 2269.

CHECK ANALYSIS, VARIATION LIMIT, REMAINDER, OTHER ELEMENTS EACH (MAXIMUM), OTHER ELEMENTS TOTAL (MAXIMUM).

5.1.4 Self-Calibrating Tests: Equipment shall have demonstrated statistical capability (precision) for each alloy family to be tested.

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5.2 Test Methods and Procedures

5.2.1 Source of Chemical Test Procedures: Methods and procedures for chemical analysis shall be as called out by the material specification. If none are specified, the applicable ASTM Method or Procedure from Volume 3.05 "Chemical Analysis of Metals and Metal Bearing Ores" shall be used. Gas fusion method shall be used for test codes G1-G5, or other methods as approved by the customer and verified by round robin and proficiency testing.

5.2.2 Working Procedures: Written procedures are required which are sufficiently detailed for each type and sequence of analysis to ensure accurate and repeatable results.

5.3 Chemistry Laboratory Matrix

A Matrix located in checklist AC7101/2 (Figure 1) shall be completed for each family of tests conducted by the laboratory. The matrix shall evidence capabilities/precision data (e.g.; detection levels, range capacities, etc.). The data used to formulate the matrix shall be generated by the laboratory, using the applicable equipment, and shall be documented. The use of manufacturer's data is not permitted.

5.3.1 Analytical calibration curves shall be traceable to NIST, foreign standards institutes, natural physical constants, or generated or derived from standardized laboratory methods using multiple laboratory analysis programs.

5.4 Personnel

Personnel shall be trained commensurate with their degree of control over testing. Required training shall be documented via the Quality Manual.

5.4.1 Operators of laboratory instrumentation shall have sufficient training to recognize proper versus improper operation of the equipment.

5.4.1a Operators shall be trained to recognize valid versus invalid data obtained from a properly executed analysis.

5.4.2 Laboratory Management shall be responsible for completing or assigning the following functions:

- a. Review and approval of data to be analyzed.
- b. Wet chemical analysis.
- c. Interpretation of data from equipment that does not provide direct numerical read-outs.
- d. Authorization of re-testing for nonconforming test values.
- e. Preparation or approval of detailed testing procedures (other than those issued by the equipment manufacturer).
- f. The above functions are performed by a degreed chemist or equivalent.

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5.5 Standards and Reagents - Chemical Testing

5.5.1 Analytical methods shall utilize reference materials which have documented traceability of analysis. Non-certified reference materials require documented multiple analyses against certified reference materials or wet chemical analysis.

Calibration for testing shall be established using reference materials, preferably certified reference materials or standard reference materials. All standards used for calibration shall be traceable to NIST, or other nationally or internationally recognized sources.

For analysis methods that do not use analytical curves, calibration is required before each test using Certified Reference Materials.

For analysis methods using analytical curves, verification of curves shall be performed periodically to assure optimum analytical precision. Documentation shall be maintained supporting the frequency of verification activities.

Frequency of drift corrections shall be documented for each instrument that utilizes this technique. Documentation shall be maintained supporting the frequency of established drift corrections.

5.5.1a CALIBRATION REFERENCE MATERIALS DEFINITIONS

- a. Reference Material (RM): A material of definite composition that closely resembles in chemical and physical nature the material which the analyst expects to analyze, and is employed for calibration or standardization. Requires documented analysis against CRMs.
- b. Certified Reference Material (CRM): A reference material, the composition or properties of which are certified by a recognized standardizing agency or group.
- c. Standard Reference Material (SRM): A certified reference material issued by the National Institute of Standards and Technology (NIST).

5.5.2 Analytical reagents which take part in a reaction or end up in the analyzed sample, e.g., solvents and diluents, shall be of the highest quality necessary to ensure the integrity of the analysis results. Consumable reagents shall be changed on a scheduled basis, to avoid adverse impact on analytical precision.

5.5.3 The laboratory shall have a procedure addressing standards having a shelf life. Standards having a shelf life shall be properly labeled to ensure they are not used after the expiration date. Shelf life disciplines shall be documented and maintained for standards susceptible to deterioration (e.g., evaporation of liquid standards, reaction with glass storage container).

5.5.4 The laboratory shall have appropriate support equipment, such as volumetric flasks, graduated cylinders, analytical balance, etc.

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- 5.5.5 The laboratory shall have standards that encompass their anticipated analytical range.
- 5.5.6 Sample preparation procedures shall be detailed enough to provide for consistency among multiple technicians.
- 5.6 Test Specimens
- 5.6.1 Chemical Specimen Preparation: Written procedures shall be used which clearly define the proper preparation technique to avoid contamination or loss of sample integrity, and to provide for consistency among technicians.
- 5.6.2 Hydrogen in Titanium: Unless the laboratory is informed otherwise by its customer, specimens shall be taken to include the processed surface(s).
- 5.6.2a Preparation procedures for hydrogen shall provide for avoidance of overheating of samples.
- 5.7 Rounding
- An observed value or a calculated value shall be rounded off "to the nearest unit" in the last right hand place of numbers used by the material specification in expressing the limiting value, in accordance with ASTM E 29. Exception: for trace elements, an observed value may be reported to the precision level of the analysis method used.
- 5.8 Re-Testing
- 5.8.1 Nonconforming Chemical Test Values: When the original test value does not conform to requirements, and the material specification does not address retesting, three (3) retests shall be performed, and evaluated in accordance with Appendix B of this standard. All specimens shall be logged, and all results recorded.
- 5.9 Check Analysis
- The customer shall define the specification used for material certification. Check limits may not be used by the laboratory to extend specification acceptance limits. If requested by the customer, a comparison to check analysis limits of the appropriate specification may be made, but the certificate must contain the following statement:
- "The material failed in accordance with paragraph X, of specification X, revision X, but is within tolerance of the check analysis limits of the specification."
- 5.10 Equipment Calibration
- Calibration requirements and methods shall be documented and referenced in the Quality Manual. Calibration frequency, standardization, and requirements shall meet the minimum requirements of Appendix D.

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- 5.10.1 Where the lower analytical limit is established by a “blank” sample, the instrument blank setting shall be verified as part of the calibration.
- 5.11 Chemical Round Robin Testing
- Round Robin testing shall be conducted in accordance with Appendix E.
- 5.12 “All Other” Elements - Residual and Trace Elements
- 5.12.1 Qualification to Trace Element Analysis requires demonstration of ability to analyze to the limits of AMS 2280 (as called out by the material specification).
- 5.12.2 Required Analyses: Analysis for specific residual and trace elements is required when called out by supplemental specifications referenced in the material specification (ref. AMS 2280), and when the material supplier informs the laboratory of known or suspected residual and trace elements (ref. Appendix A).
- 5.12.3 Acceptance of Unspecified Elements: Analysis is not required for such elements except as described above. If, however, analysis is performed and such elements are found, reporting and acceptance shall be as described below.
- a. If the specification has allowable limits for “All Other” elements, acceptance shall be to these limits (Such notes will be present in most titanium alloy specifications). Elements having specific percents determined should be reported.
 - b. Other elements found need not be reported if they do not exceed the following limits:

All Alloy Families: Elements reported as “< 0.xxx”, where “0.xxx” represents the minimum (calibrated) detection limit of the test method. For such cases, when this minimum detection limit is higher than 0.001 and the element is detected, it should be recorded in the laboratory’s analysis records.

Iron, Nickel, Cobalt Alloys: (Maximum for each element and maximum total of all elements within that group.)

<u>Total</u>	<u>Each</u>
----	0.005% B (in Fe alloys)
0.015%	0.015% B (in Ni, Co alloys), Mg
0.010%	0.05% Al, Zr
0.30%	0.15% Ti, V, Cr, Cu, Fe, Cb[Nb], Hf, Ta, W, Mo (in Ni alloys)
----	0.50% Mo (in Co alloys)
1.00%	1.00% Ni, Co
 - c. Uncalibrated (semi-quantitative) Values: If instrumental analysis detects and reports unspecified elements above background levels, but the analysis procedure is not calibrated for those elements, the laboratory shall develop a documented procedure for disposition. Procedures should be sensitive to significant trends or abnormally high levels of indicated results; data should be included in the laboratory’s records.

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5.13 Operating Parameters

The laboratory's procedures shall provide for control of operating parameters which may affect test data. Listed below are minimum requirements for the following techniques:

5.13.1 OPTICAL EMISSION SPECTROSCOPY

- a. Pre-burn.
- b. Integration time.
- c. Number of burns per sample.
- d. Burn placement.
- e. Electrode selection, preparation, and cleaning.
- f. Sample/standard surface preparation.

5.13.2 SOLUTION ANALYSIS (AA/ICP/DCP)

- a. Graphite furnace/drying temperatures.
- b. Interference lines and matrix effects.
- c. Verification of linearity of two (2) point curves.
- d. Drift correction per ASTM-E-1097 (ICP/DCP).

5.13.3 X-RAY FLUORESCENCE

- a. Samples and standards are prepared in the same manner.
- b. Surface finish is controlled, as applicable to the longer wavelength elements.

5.14 Test Records and Certifications

The test records shall reflect the standards used, and traceability to the analytical curve. The test certificate shall list the method of analysis.

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6. MECHANICAL TESTING:

6.1 Test Types, Codes, Methods and Procedures

Codes are as follows for mechanical property tests. The noted test specifications and standards apply unless otherwise specified or approved.

<u>Code</u>	<u>Test Type</u>	<u>Test Specification/Standard</u>
A	ROOM TEMPERATURE TENSILE	ASTM E 8
B	ELEVATED TEMPERATURE TENSILE	ASTM E 21
C	STRESS RUPTURE	ASTM E 139 (Smooth), ASTM E 292 (Notched)
XA	CREEP	ASTM E 139
N	IMPACT TESTING	ASTM E 23
O	HIGH CYCLE FATIGUE (HCF)	ASTM E 466
P	FRACTURE TOUGHNESS	ASTM E 399
Y	LOW CYCLE FATIGUE (LCF)	ASTM E 606
XE	CRACK PROPAGATION MEASUREMENT	ASTM E 647
XH	CYCLIC RUPTURE	ASTM E 139
XN	BEND TEST	ASTM E 290

COATINGS (AC7109/5)
 MECHANICAL THICKNESS
 BOND STRENGTH
 1. Tensile (ASTM C633)
 2. Bend Testing

6.2 Mechanical Testing Procedures

Written procedures are required which are sufficiently detailed to be followed for the specific test equipment to be used and specific test to be performed, and shall conform to the applicable ASTM test method.

6.3 Mechanical Testing Laboratory Matrix

A matrix in checklist AC7101/3 (Figure 1) shall be completed for applicable tests, and shall include reference to applicable Round Robin programs.

6.4 Mechanical Testing Equipment

It is preferred that the tensile testing equipment automatically plots the stress/strain curve and maintains a constant strain rate. When this is not done, the laboratory shall have documented substantiation for the alternate method used. Tensile machines shall be equipped with a method of strain rate control.

6.4.1 Calibration Frequencies shall be per Appendix D unless otherwise noted.

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- 6.4.2 Tensile, Stress Rupture, and Creep test machines shall be calibrated per ASTM E 4; calibration equipment shall be per ASTM E 74.
- 6.4.3 Extensometers shall conform to ASTM E 83, Class B-2 or better. Calibration shall be per ASTM E 83 or equivalent. Extensometers shall be calibrated for a particular machine and electronics set-up.
- 6.4.4 Temperature Measuring Devices shall be either a Group B precision potentiometer or alternate device calibrated and traceable to a NIST standard.
- 6.4.5 Specimen Furnaces shall be resistance, circulating hot air, or other types and approved during qualification via documentation per matrix located in the applicable checklist. Induction heating shall not be used for tensile, stress rupture, or creep tests unless permitted by the customer; for other tests, calibration studies shall demonstrate that differences in response between the thermocouple and specimen do not introduce temperature errors. Furnace design and practice shall prevent over-temperature outside the measured section. A sufficient number of thermocouples shall be used to ensure that the temperature gradient requirements of ASTM E 21 are satisfied.
- 6.4.6 Loading Weights shall be calibrated to produce a loading accuracy (including leverage effects if applicable) of 0.5% or better. Re-calibration frequency shall be per Appendix D, Note (b). Weights which are checked on a calibrated scale prior to each use do not require a separate calibration program.
- 6.4.7 Calibration and Maintenance Records: Applicable information shall be supplied on the matrix. Records shall include:
- a. Machine identity, including type, manufacturer's name, serial number, and capabilities or capacity.
 - b. Calibrating procedure. Standards used to calibrate instruments must be traceable to NIST. Calibration equipment records shall include type, manufacturer's name, serial number, date of last calibration and next scheduled calibration, and accuracy.
 - c. Name and address of the calibrating organization.
- 6.4.8 If testing is interrupted due to power outage, equipment failure, etc., test records shall document the following: Date and time interruption occurred, equipment malfunction explanation, current status of test, data recorded prior to interruption, corrective action taken, if required.
- The laboratory shall notify customer(s) of test interruption as applicable.
- 6.5 Thermocouples (T/C) shall be selected in accordance with customer criteria. T/Cs used for elevated temperature tests shall be reported. Prior to re-use of any T/C, kinked wire shall be removed and that junction replaced; all obviously oxidized or damaged junctions shall be removed and replaced. There shall be no twisting of the wires prior to the junction point. If thermocouples are attached by tack welding, they are to be located in the radius interface, and not in the test section. The laboratory shall have a procedure addressing reuse of thermocouples.

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- 6.5.1 T/C calibration: Wire shall be calibrated to the requirements of ASTM E 220 (including limits of error) at the maximum and minimum temperatures, and at the maximum intervals given below. Individual T/Cs may be calibrated in lieu of wire calibration. The calibration procedure of the laboratory or outside agency shall be documented.

INTERVALS

200°F (111°C): Types "R" and "S"

50°F (28°C): Types "N" above 1000°F (538°C)

50°F (28°C): All other types (except "K" as required below)

- 6.5.2 Type "K" Thermocouple Calibration

- a. Only "premium" grade wire is permitted.
- b. Each spool shall be certified at both ends and at 2000 feet intervals, per ASTM E 220 (including limits of error) at the maximum and minimum temperatures.
- c. A certification data point within 150°F of the test temperature shall be required for test temperatures between 950°F and 1550°F. For example, a T/C certification at 1100°F and 1400°F will allow the T/C to be used for test temperatures from 950°F to 1550°F.
- d. A certification point within 50°F is required for temperatures below 950°F and above 1550°F.

- 6.5.3 Type "K" Thermocouple Restrictions: Thermocouples may not be re-used unless all wire previously exposed at temperatures above 1000°F (538°C) has been removed and a new junction made.

- a. Type "K" may not be used for testing above 1800°F (982°C) unless the laboratory has demonstrated wire stability in the testing environment (typically thin T/C). Wire is subject to rapid deterioration. Stability is defined as thermocouple performance per ASTM E 220 (premium wire) for the test duration.
- b. Thermocouple Junctions: Above 1600 °F (870 °C), compression bonded junctions are preferred; welded junctions may be used if data for the welding process used demonstrates that progressive corrosion of the joint does not result in drift outside the limits noted below. (Note: Welded junctions in Types "R" and "S" may be used at any temperature.)
- c. Wire stability for creep and stress rupture testing above 1600°F (870°C) shall be determined by the comparison method of ASTM E 220. The exposure temperature must equal or exceed the maximum temperature at which the wire will be used; exposure time shall be 25 hours minimum. The wire is acceptable provided the indicated temperature does not drift more than 5°F (3°C) during exposure.

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6.6 Mechanical Testing Specimens

6.6.1 Configuration shall be in accordance with the applicable test method/procedure. If not specified, the laboratory may select specimen configuration for requested test which is documented in its internal manual. Specimen drawing shall be recorded.

6.6.2 Preparation: Specimen preparation shall be as called out in Section 10. The laboratory shall ensure that specimen configuration and preparation procedures are documented. All specimens are prepared to laboratory or customer drawings. If a preparation is done by an outside source, the outside source shall be identified, and is an approved subcontractor to the laboratory.

6.6.3 Specimen Dimension Control: Statistical Process Control shall be used by the testing facility when 100% measurement is not used for gage section dimensions or other dimensions which might affect testing axiality or stress concentration. Examples: uniformity, concentricity, required taper, undercut of radii at gage section.

Test specimens shall be visually inspected by the testing function, for surface finish, and surface damage prior to the test, and if a magnification other than 1X is used, it shall be recorded. When NDT methods are used, they are to be noted as well. Any observed surface irregularities shall be listed in the test record, if the test is to still be performed.

Notch surfaces are to be inspected at a minimum of 10X magnification.

6.7 Specimen Heat Treatment

Heat treating of material to be tested requires qualification to laboratory testing code "XG". The specific specimen heat treating method used shall be noted on the certificate.

6.8 Thermocouple Attachment

Thermocouple attachment by spot welding directly on the reduced section of the specimen is prohibited. Methods of attachment are test type and material type dependent. The T/C junction shall be in intimate contact with the test specimen. Procedures shall identify standard attachment practice.

6.9 Extensometer Attachment

Extensometers shall be attached directly to the reduced section of the test sample. Standard specimens with integral external notched lips at the ends of the reduced section (shoulders) to prevent slippage are permitted, provided that correction factors are used. Extensometers attached to grips, pull bars, or the load train assembly shall not be used for required measurements. Extensometer attachment practices are test type and material type dependent. Procedures shall identify attachment practices.

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6.10 Alignment Capability

Calibration of alignment capability per Appendix D is required for tests requiring axial loading. Calibration of elevated temperature test equipment may be performed at room temperature due to strain gage temperature limitations. For assurance of alignment disciplines, and analysis of test results, reference ASTM E 1012. For additional information; however, requirements of this specification take precedence.

6.10.1 Required Capability: Maximum bending capability shall be demonstrated as follows: ("Brittle materials" are those with expected percent elongations less than five percent in 4D. Reference ASTM E 606 for techniques).

Static Tests: 10% (non-brittle materials)
8% (brittle materials)

Cyclic Tests: 5%

Tests, as defined by the customer, requiring specific maximum bending strain (i.e., specific equipment and fixtures) shall require alignment verification immediately prior to test and demonstrate repeatable capability to the above limits.

Percent bending strain is the greatest difference in axial strain under load between the average strain (of 3 or 4 strain gages in any circumferential set) and any individual gage in that set, divided by the average strain, times 100. Percent bending strain shall be calculated in accordance with ASTM E 1012.

6.10.2 Loads During Evaluation: For the test conditions described below, "lowest maximum load" is as follows, considering the material strengths and specimen cross-sections to be evaluated.

- Tensile tests: Lowest load for required yield strength.
- Constant load tests: Lowest load for producing specified stress.

Cyclic tests: Load representing the minimum stress or strain, tensile and/or compressive, specified for the test.

6.10.3 Static Test Equipment: Test set-up shall include the test machine, grips, and selected pull train. Strain readings shall be taken at zero load, at the lowest maximum load of the set-up for which the machine is being qualified, and two step loads at higher values.

6.10.4 Cyclic Test Equipment: Test set-up shall include the test machine and grips. Calibration is performed under static load conditions, but must include two or more test repetitions. Strain readings shall be taken at zero load, at the lowest maximum load/strain (both tensile and, if used, compressive) of the set-up for which the machine is being qualified, and two step loads at higher values.

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6.10.5 Calibration Specimen(s): Bars shall represent the minimum and typical reduced section lengths to be tested. A strain gage set comprises axial strain gages located at either 90° or 120° intervals around the circumference of the specimen reduced section. The number of sets is dependent on the length of the reduced sections, as follows:

- a. Specimens with reduced sections of 0.75 in (19 mm) or shorter: One set at center.
- b. Specimens with reduced sections longer than 0.75 in (19 mm): One set each at center and each end.

6.11 Replacement Mechanical Property Tests

Specimens and test set-ups shall always be visually checked for damage prior to testing, to preclude subsequent replacement of tests insofar as possible. Accepted reasons for replacement of mechanical tests are as follows:

- a. Original specimen had a poorly machined surface or had other visible surface damage prior to testing.
- b. Original specimen had incorrect dimensions.
- c. Incorrect preparation procedures were used which may have affected properties (e.g., grinding without lubricant).
- d. The test procedure was incorrect, or was performed incorrectly.
- e. Failure of mechanical test specimens occurred outside the reduced area or gage length.
- f. For elongation determinations, the fracture was outside the middle half of the gage length.
- g. Malfunction of the testing equipment, including thermocouple malfunctions.
- h. Improper heat treatment of the specimen, when the specimen heat treatment does not represent the product condition ordered (e.g., "capability" tests on aged specimens for products which are ordered in the annealed condition).

These conditions shall be reason to replace either conforming or non-conforming test results, and shall not be used as a convenient way to conduct replacement tests when non-conforming conditions exist.

6.12 Re-Testing

Re-testing (as opposed to replacement testing) for product acceptance is permitted per Appendix B (three re-tests) or as authorized by the material specification or other contractual document. Nonconformance of a re-test specimen precludes further re-testing. All specimens shall be logged and all results recorded; analysis of results and reporting results on the Certification shall be in accordance with Appendix B.

6.12.1 Re-tests for Information: After material has been found to be nonconforming, additional re-testing may be performed at the option of the material supplier and results reported in the nonconformance document, as an aid for disposition of the nonconforming condition.

6.13 Round Robin Testing - Mechanical Properties

Round Robin testing shall be conducted in accordance with Appendix E.

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6.14 Tensile Testing

All tensile tests shall be conducted in accordance with ASTM E8 or E21, unless other specification is specified.

If the specification does not reference a specific strain rate, the strain rate for both room and elevated temperature tensile testing is to be .003 to .007 inch/inch/minute through yield, and .05 inch/inch/minute after yield, with the yield point being determined at 0.2% offset, unless specified otherwise.

Percent elongation shall be measured and reported in accordance with the material specification.

For elevated temperature tensile testing, the minimum soak time at temperature shall be 30 minutes.

6.14.1 Tensile Test Records: As a minimum, test records shall include the requirements listed in ASTM E 21 or ASTM E 8, as applicable, and the following information if applicable. Upon request, records shall be furnished to the Purchaser within three working days.

- Failure location.
- Identification of equipment used including make, model and capacity of testing machine; make, gage length and class of extensometer; make and size of furnace, test temperature, type of controller, and description of thermocouple including material, wire size, attachment technique and shielding. The number of thermocouples used shall be recorded.
- All applicable test data (YTS, UTS, %EL, R/A).
- Name of individual performing test and date of test.

6.14.2 Conversion from SI (Metric) Stress Into English Units: When strength values are determined in the SI (metric) system, SI data are to be tabulated in MPa, then converted into psi per ASTM E 380 Method A. Rounding off may then be done in the English system as provided herein. Conformance shall be determined against the English limits of the specification, unless SI units are specified.

6.14.3 Rounding of Test Data

When test data is rounded, it shall be in accordance with ASTM E8, ASTM E21, and ASTM E29.

6.15 Stress Rupture / Creep - Testing (Codes C-XA)

The laboratory's procedures shall conform to ASTM E 139, and shall require that sufficient thermocouples are used to assure that the temperature gradients of ASTM E139 are met. If the laboratory temperature varies by more than $\pm 3^{\circ}\text{C}$ ($\pm 5^{\circ}\text{F}$), then data acquisition will conform to the requirements of ASTM E139. If extensometer shoulder attachment is used, the laboratory shall address this in the Quality System Documents, and provide for the use of correction factors. Specimen soak time is 1 hour after stabilization.

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6.15 Continued

For cyclic rupture, the ramp up, hold time and ramp down are to be verified using a digital timer, and the loading cycle shall be monitored once per working shift.

The notch root radius is to be measured to the nearest .0005 inch (.01 mm) prior to testing, and a smooth notch profile shall be verified using an optical comparator.

6.15.1 Creep Test Records: As a minimum, test records shall include the following information:

The make, model and capacity of the machine. Identification of temperature measuring equipment, including the controller, thermocouple wire size, type and grade, as well as the number of thermocouples, and their attachment method. The type of shielding used on the thermocouples shall be listed. The extensometer make and identification shall be recorded, along with the class, gage length and distance between and location of attachment points.

The following testing variables are to be recorded:

- a. Incremental loading curves or data showing specimen strain on loading, elastic and plastic components, zeroing the curve to exclude extraneous movement within the pull train.
- b. Tabulated creep strain vs. time data from which creep strain and time to corresponding minimum creep rate may be obtained.
- c. Time to failure or discontinuation of test.
- d. Plot of creep strain vs. time with X-axis labeled HOURS and Y-axis labeled STRAIN.
NOTE: Plots provided to the Purchaser shall be "log-log"; a combined curve of total elastic strain [(a)+(d)] is desired.
- e. Weight and lever ratios, if adjustable ratio machines are used.
- f. Time/temperature history of specimen heat-up.
- g. Ambient temperature in the laboratory.
- h. For creep, the date, time of day of each temperature measurement and extension observation.

6.15.1.1 Additional Requirements for Notched Bars

- a. Initial shoulder diameter for round specimens.
- b. Initial notch diameter.
- c. Initial notch root radius.
- d. Failure location.

6.15.2 Test certificates for creep testing shall contain the following information:

- a. Test temperature and tolerance.
- b. Applied stress.
- c. Test duration.
- d. Percent elongation (if required).
- e. Percent Reduction of area (if required).
- f. Plot of creep strain versus time (if required).
- g. Initial incremental loading curves, or data which can be added to the strain/time curve to permit calculation of total plastic deformation.

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- 6.15.3 Rounding of Stress Rupture and Creep Data: Rounding as follows may be used to report observed or calculated test values for determining conformance to specified limits.
Reference: ASTM E 29, E 139, E 292.

<u>For This Property</u>	<u>If In This Range</u>	<u>Round to Nearest</u>
Test Stress	Must be within 1% of specified.	Round to specified stress.
Time	Below specified min. time Above specified min. time	0.1 hour 1 hour
Elongation and Reduction of Area	5% or above Below 5%	whole percent half percent

6.16 Fatigue Testing - Additional Requirements (Codes Y-XH)

The following requirements are in addition to requirements of standard test methods. Modern hydraulic fatigue test machines may also be capable of performing other types of high cycle fatigue testing.

The tests shall be controlled per ASTM E 606. The temperature throughout the uniform gage section shall be maintained within 1% of the test temperature, or 2°C, whichever is greater. Extensometers shall be free from "drafts" which may affect the reading. The laboratory must have a written procedure for extensometer attachment to prevent damage or bending strain on the specimen. The hot gage length and hot area are to be used in all calculations.

The diameter of the gage section shall be measured, using a non-contacting measurement technique, prior to testing. The surface of each specimen is to be inspected at 20X magnification prior to loading, and any discrepancies shall be noted on the test certificate. The finished specimen shall be in accordance with customer specification. All specimens are to be individually protected until testing to prevent surface damage. Failed specimens are preserved to protect the fracture faces, and retained for 1 year minimum, if not returned to the customer.

Strain ratios and cyclic frequencies shall be in accordance with specimen drawing or material specification.

- 6.16.1 Facility Temperature and Humidity: Temperature and humidity shall be measured and recorded continually. The temperature of the test facility shall be maintained in the range of 15-29°C (59-84°F). More stringent limits shall be used if necessary to ensure consistent operation of equipment.

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- 6.16.2 LCF Equipment: Each test machine shall be capable of controlling wave shape, and shall provide for and guarantee limits for at least one of the following: piston displacement, force, specimen displacement. The testing machine shall be calibrated to control strain within 1% of the range between the maximum and minimum control limits, and load within 1% of the difference between the maximum and minimum control limits. The test stand shall be capable of detecting and monitoring crack initiation. Extensometers shall be a minimum of Class B-2.

Machine alignment shall be within 5%, and shall be repeatable.

- 6.16.3 Test Continuation and Failed Specimen Retention: Fatigue tests shall be continued until failure unless otherwise specified. Upon completion of testing, failed bars shall be preserved to protect the fracture faces and returned to the customer as required.

- 6.16.4 LCF, Strain Control to Load Control: Tests shall be run in strain control mode until failure. Switching from Strain Control mode to Load Control is permitted only when authorized by the appropriate material specification, part specification, or written customer direction. The laboratory shall comply with conditions established for switching, and shall have an internal procedure addressing this function. Strain control switching shall be listed on the certificate.

- 6.17 Cyclic Rupture Testing - Additional Requirements (Code XH)

- 6.17.1 Testing shall be performed to the requirements of this standard, and the applicable Material Specification.

- 6.17.2 Cycle Times: Ramp up, hold time, and ramp down shall be verified using a digital timer.

- 6.17.3 Temperature and cycle time monitoring shall be at 8 hour (maximum) intervals. Checks should be made more frequently during the first day.

- 6.17.4 Test Report - Cyclic Rupture shall, as a minimum, include the applicable requirements listed in ASTM E139.

- 6.17.5 Round Robin programs shall be established in accordance with Appendix E.

- 6.18 Impact Testing

- 6.18.1 Impact testing shall be conducted to ASTM E 23, unless specified by the customer.

- 6.18.2 Notched-bar impact testing shall be completed by either the Charpy or the Izod method.

- 6.18.3 The test report shall contain the following information:

- a. Specimen type and size, if not standard.
- b. Test temperature.
- c. Absorbed energy.
- d. Lateral expansion, if required.
- e. Fracture appearance, if required (% shear fracture).

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6.19 Fracture Toughness

6.19.1 Fracture toughness testing should be conducted to ASTM E 399, unless specified by the customer.

6.20 Crack Propagation

6.20.1 Crack propagation testing should be conducted to ASTM E 647, unless specified by the customer.

6.21 Bend Testing

6.21.1 Bend testing should be conducted to ASTM E 290, unless specified by the customer.

7. METALLOGRAPHY AND MICROHARDNESS:

7.1 Test Types - Codes

Codes are as follows:

L METALLOGRAPHY (GENERAL MICRO)

L1 MICROHARDNESS (INTERIOR)

XL METALLOGRAPHY (MACRO)

LS MICRO: SURFACE CONDITIONS

L2 ALLOY DEPLETION

L3 OXIDATION/CORROSION

L4 CASTING (MOLD) REACTIONS

L5 MICROHARDNESS (SURFACE)

L6 DIFFUSION COATINGS

L7 IGA, IGO

L8 ALPHA CASE: WROUGHT TITANIUM

L9 ALPHA CASE: CAST TITANIUM

COATINGS (AC7109/5)

METALLOGRAPHY

1. High/Low Temperature Hard Coats

2. Anti-Fretting Coatings

3. Thermal Barrier

4. Abrasive Seal

5. Abradable

6. Dimensional Build-up

7. High Temperature (LPPS)

8. Diffusion Coating

9. PVD Coatings

THICKNESS

Metallographic

MICROHARDNESS

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- 7.1.1 Metallographic Laboratory Matrix: A matrix shall be completed for applicable tests, including reference to all specific evaluations which are required by this standard to be referenced.
- 7.1.2 Code "L1" MICROHARDNESS (ASTM E 384) applies to microhardnesses at applied loads less than 1 kgf performed on mounted and metallographically polished specimens. Test Code "M" HARDNESS includes Vickers hardness (ASTM E 92) at applied loads of 1 kgf to 120 kgf.
- 7.1.3 Test Code "XL" (Macro) is intended to apply to macroetch of material sections (as would be performed by a laboratory).
- 7.1.4 Metallographic and microhardness testing by other sources (e.g., heat treat, plating, metal spray, diffusion coating sources) may be approved and documented for the applicable Special process.
- 7.2 Test Methods and Procedures
- Methods and procedures for metallographic testing and examination shall be as called out by the material specification.
- Detailed written procedures for preparation and etching of specimens and required metallographic comparison standards as required for proper evaluation shall be convenient to the operator.
- The following ASTM Methods shall be used as a reference in preparing detailed procedures:
- ASTM E3 -- Preparation of metallographic specimens
 - ASTM E407 -- Microetching of metallographic specimens
 - ASTM E340 -- Macroetching of metallographic specimens
- For Microscopic evaluations, a written procedure shall cover selection of the area to be evaluated, and measurement techniques for surface layer evaluations, which shall address low magnification scans to locate area of concern, and then examination at the required magnification. If grain size measurement are in accordance with ASTM E112, and Titanium microstructures are examined in accordance with AMS 2380, a evaluation of the general microstructure is mandatory.
- 7.3 Metallographic Personnel
- All personnel shall be trained in accordance with a written plan, which shall include general and specific aspects of the procedure to be performed. Operators shall be trained to recognize proper operation of equipment.
- Laboratory management shall assign responsibility for review and approval of tests, training and qualification of technicians, standards and procedures.

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- 7.3.1 Degreed metallurgists or equivalent are required for the approval of acceptance standards generated by the laboratory. "Equivalency" requires formal training in metallurgy plus extensive applicable experience and shall be controlled by the Quality System Documents.
- 7.4 Equipment and Calibration
- Microscope design shall satisfy the minimum equipment requirements outlined by the standard test specification.
- Dimensional measurement calibration of filar eye-pieces is to be done against stage micrometers and shall be documented. Special eye-pieces used for grain size determination shall be calibrated and documented.
- Video equipment used for numerical evaluations is to be verified at the time of evaluation as a system, to ensure the full screen evaluation is correct. Records of verification shall be maintained in a log book, or equivalent.
- 7.5 Specimen Material Supply
- The laboratory shall maintain a supply of materials or specimens for the purpose of operator training in preparation and interpretation and for metallographic process verification. Alloys shall be of the appropriate type and form (wrought or cast) as alloys generally examined; photographic standards may be used for interpretation. A written procedure shall be maintained which describes standards and their use.
- 7.6 Specimen/Surface Preparation
- The method of sectioning the sample shall be such that it does not create surface damage to the specimen which could lead to errors in evaluation. A diamond wheel, using a slow cutting rate, or a fluid-cooled wheel which enters, but does not exit the piece, may be used for this purpose. Sufficient material removal may also be accomplished by polishing after cutting.
- Preparation method procedures shall ensure perpendicularity suitable for measurement of surface condition dimensions in accordance with applicable specifications. Mounting practices shall not damage the surface to be evaluated. Specific surfaces to be evaluated are to be identified by the metallographer during the examination.
- If multiple specimens are contained in the same mount, provisions are made to ensure positive identification of each specimen.
- Surface preparation method(s) shall produce a uniform texture. The prepared specimen shall show no evidence of smearing, machining or grinding marks, or overheating from preparation (either before or after etching).

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7.6 Continued

Electropolish methods may be used for removal of mechanically stressed surface layers prior to mechanical polishing. When used for final polishing prior to etching, electropolished surfaces shall be sufficiently free from scratches and pits (prior to etch) to permit proper evaluation (before or after etch, as required.) Electropolishing shall not be used when microexamination of edges is required.

7.7 Etching

7.7.1 Etchant Control: Any container of an etchant which is to be stored (i.e., not used immediately) or re-used shall be identified with regard to composition, make-up date, shelf life and storage considerations, and precautions for use. Macroetch tanks, if used, shall be controlled by chemical analysis and/or etching response on known material.

7.7.2 Fixed Etching Practices: The etching method shall be selected and described with respect to its purpose (e.g., segregation, grain flow, relative grain size). As a minimum, procedures shall define the following: intended purposes, material surface preparation, etchant types and concentrations, material and etchant temperatures, etchant time and application, agitation, rinsing and drying, electrical current and voltage (if used), known defect and comparison standards. Changes in procedure shall be documented, with reason for change.

7.7.3 Etched Surface Appearance: Zones to be evaluated shall be essentially free from evidence of mechanical stress from specimen preparation, and from staining and water spots.

7.8 Metallographic Standards and Photographs

7.8.1 Standards: The laboratory shall maintain controlled metallographic standards, which are convenient to the operator, for evaluations to be performed under contract requirements. These standards are to be controlled by written procedures regarding their establishment and usage. Standards may be photographs, specimens, or other representations (e.g., microscope reticules for grain size measurements). When standards are not established by the material specification or by the material supplier, initial or supplemental standards may be issued by the laboratory at the time the evaluation is performed. These standards shall be approved by the chief metallurgist or equivalent.

7.8.2 Photographs (including replicas) provide permanent records of the material evaluated. When made, they shall be identified with respect to the job, specimen number, material location represented, magnification, and etchant used.

7.8.3 Metallographic features not covered by acceptance standards shall be referred to a metallurgist, or equivalent.

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7.9 Microexamination at Near-Surface Sections and Microhardness

Typical surface conditions requiring special precautions during evaluation include those listed below.

- a. Alloy depletion
- b. Oxidation/corrosion layers, including alpha case
- c. Casting surface reactions (mold reactions)
- d. Microhardness: Vickers (Diamond Pyramid Hardness: DPH), Knoop
- e. Diffusion coatings (e.g., carburizing, nitriding)
- f. Intergranular Attack (IGA), Intergranular Oxidation (IGO)
- g. Alpha Case: Wrought
- h. Alpha Case: Cast

7.9.1 The following requirements apply to the applicable surface condition:

A. Alloy Depletion

The laboratory shall have an internal procedure for alloy depletion evaluation, which identifies selection of specimen areas to be evaluated, as well as measurement techniques. The procedure must dictate scanning entire surface, at low magnification, to locate areas of maximum depletion, then require examination of these areas at the specified magnification. The absolute maximum depth is to be reported, and grain boundary effects are considered in the examination.

The etching procedure is to be alloy-specific, and address alloy form (cast vs. wrought). The heat treat condition shall also be taken into account.

Representative specimens, with known alloy depiction are to be used for both training, and etching verification purposes.

B. Oxidation/Corrosion Layers

The laboratory shall have an internal procedure for corrosion/oxidation layers, that ensures retention of layers to be evaluated, as well as measurement techniques. This procedure may include provisions for low magnification examination prior to mounting, polishing to remove cutting damage, and/or surface coating (plating) to protect the layer. Mounting prior to sectioning, to facilitate obtaining the specific area of examination.

Procedures shall define whether the examination is to be performed in the etched and/or un-etched condition. Initial examinations are done in the un-etched condition to avoid preferential attack which may remove the affected layer. The procedure must dictate scanning the entire surface at low magnification, to locate areas of maximum corrosion/oxidation, then require examination of these areas at the specified magnification. The absolute maximum depth is to be reported.

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7.9.1 Continued

C. Casting Mold Reaction Layers

The laboratory shall have an internal procedure for casting mold reaction layers, as well as measurement techniques. The procedure must dictate scanning entire surface, at low magnification, to locate areas of maximum depth of mold reaction, then require examination of these areas at the specified magnification. The absolute maximum depth is to be reported.

The etching procedure must be alloy-specific and address alloy form (e.g., cast or wrought). The heat treat condition shall also be taken into account.

Representative specimens with known surface conditions are to be used for both training and etching verification purposes.

D. Microhardness Testing

Microhardness testing shall be performed in accordance with ASTM E 384, in an environment that is essentially free from vibration. Test specimen surface finish is to be sufficiently polished to provide a good contrast of the perimeter of the indentation against the specimen surface. Specimen flatness and alignment are to be such that indentations closest to the edge are clearly defined, and that the length of the legs of the diagonals are within 20% of each other for Knoop, and that both legs of the same diagonal are not noticeably different in length for Vickers. Indentations for Vickers hardness testing, which are closer to the edge than the length of the indentation diagonal perpendicular to the edge, are to be disregarded. At least two (2) indentations shall be made on the test block and compared for repeatability.

Written procedures shall cover the selection of the area of the specimen to be examined.

There shall be no indication of metal distortion produced by specimen preparation. If specimens are etched, the etch is to be light enough so that the indentations remain clearly defined.

Equipment shall be verified to ASTM E 384, and shall be dictated by a written procedure. Verification is to be performed using five (5) indentations, with repeatability and maximum error conforming to ASTM E 384. Hardness test blocks used for verification shall be certified for mean hardness number, test load and magnification. The block shall be identified with a serial number, traceable to the certifying agency.

Prior to taking measurement indentations, two (2) preliminary indentations shall be made and compared for repeatability to ensure proper seating of the specimen. If microhardness values are converted to other scales, the laboratory shall have data available for each alloy and hardness range. The test load shall be up to 1 kgf and shall be reported. Material homogeneity shall be considered when selecting test load. The test load application and removal is done smoothly, and shall be applied for a period of 10-15 s, unless required otherwise by the product specification. Non-standard durations shall be documented.

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7.9.1 Continued

E. Diffusion Coatings

The laboratory shall have an internal procedure for diffusion coating that ensures retention of layers to be evaluated, as well as measurement techniques. This procedure may include provisions for low magnification examination prior to mounting, polishing to remove cutting damage, and/or surface coating (plating) to protect the layer, and mounting prior to sectioning to facilitate obtaining the specific area of examination.

Procedures shall define whether the examination is to be performed in the etched, and/or un-etched condition. Initial examinations are done in the un-etched condition, to avoid preferential attack which may remove the affected layer. The procedure must dictate scanning entire surface at low magnification to locate areas of maximum corrosion/oxidation, then require examination of these areas at the specified magnification. The absolute minimum and/or maximum depth at any one point is to be reported.

F. Intergranular Attack - Intergranular Oxidation (IGA - IGO)

Examinations for IGA/IGO are to be performed in accordance with customer specifications in the as-polished condition, unless directed otherwise. The laboratory shall have a written procedure that covers selection of the specimen areas to be evaluated, which provides for scanning of the entire surface at low magnification, to locate the areas of maximum IGA/IGO. The procedure must address measurement techniques, and specify that absolute maximum depth is reported.

G. Alpha Case on Titanium Alloys (Cast/Wrought/Thermal Processes)

Written specimen preparation procedures shall ensure retention of surface layers to be evaluated, and provisions made in the procedure, to utilize at least one of the following methods of preservation:

1. Edges examined at low magnification prior to mounting.
2. Sufficient polishing to remove layer damage by cutting.
3. After mounting, view at 50X to confirm lack of edge gap.
4. Surface coating (plating) applied over surface to be examined.
5. Sample mounted-sectioned-remounted and polished.

Cast and wrought material, with known presence of alpha case, is to be used for developing etch procedure. Each etchant is to be stored in acid-resistant container, not glass, and shall not be returned to the container after usage. Etchant containers are to be labeled with the date of preparation, and etchant shall be discarded when it fails to reveal case on the "known" specimen.

Etching is to be performed to the laboratory's procedure, or customer requirement, until white layer appears or over-etch occurs. This may be accomplished by either the two-step, or one-step etch process, as defined in Table 5 of AC7101/4. The "known" specimen shall be run at the beginning and end of each process batch. If it fails to yield the white layer, the etchant is to be discarded, and all specimens re-etched and evaluated with a fresh etchant.

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7.9.1 Continued

After etching, the entire edge of the specimen shall be scanned at 40-50X, 100X or 500X, as required to locate the white layer. The maximum depth is to be determined at 400-500X, or in accordance with customer requirements, and the case depth is to be reported to the nearest .001 inch (cast or wrought product), .0001 inch for thermal processes, or in accordance with customer requirements.

When bend testing is required, the minimum angle shall be 105° around a diameter of 10 times the nominal thickness of the specimen.

The auditor shall witness the processing of alpha case specimens with "known" case for cast, wrought and thermal processes, as part of the audit of metallography.

7.9.1.1 Carbide Inclusion Rating: The laboratory shall have specific charts or plates for Method D of ASTM E45, for materials with expected low inclusion content. A detailed written procedure shall establish specific preparation and examination practices.

7.9.2 Edge Retention: Specimens shall be cut, mounted, and polished to produce a surface cross-section examination zone that is free from distortion of the metal structure and within the same focal plane at the magnifications required. There shall be no gap between the surface and the mounting material that would subsequently result in bleed-out of etchant.

7.9.3 Surface Layer Etching: Because ASTM E407 etching practices are not generally designed for evaluating surface reaction effects, the laboratory must have its own detailed procedures for any such evaluation it performs. Specimens with known surface conditions of the type to be evaluated shall be available for personnel training and process verification.

7.9.4 Magnification: Minimum magnification for evaluation shall be:

- Layers up to 0.001 in (0.03 mm) 400X
- Layers thicker than 0.001 in (0.03 mm) 100X
- Layers thicker than 0.010 in (0.25 mm) 40X

7.10 Micro Evaluation

A written procedure and/or customer specification shall cover selection of the specimen areas to be evaluated, and measurement techniques for surface layers. For example: (1) For grain size, scan the entire specimen at low magnification and select a "typical" and "extreme" area for detailed characterization; (2) For surface layer thickness, examine 5 to 10 areas at random and report minimum-maximum-average.

7.11 Macro Examination of Cross Sections

7.11.1 Surface Preparation: Method(s) used shall produce a uniform texture, free from surface conditions (e.g., grind marks, stresses, overheating) that could affect etching response or interpretation. Chemical methods may be used to remove mechanically-stressed surface layers prior to etching.

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7.11.2 Macro Etching: The etching method shall be selected and described with respect to its purpose. For example: detection of chemical segregation, grain flow, relative grain size.

7.12 Round Robin

Round Robin testing shall be conducted in accordance with Appendix E.

7.13 Certificate/Test Report

In addition to information required by Section 4 and the applicable specification, the Certification shall describe:

- a. Etchant used.
- b. Magnification used.
- c. Specimen number.
- d. Material location represented.
- e. Characteristic(s) evaluated.
- f. One of the following:
 - Numerical values (e.g., grain size).
 - Reference to established standards (e.g., specification microstructure levels).
 - Specimens or photographs (including replicas) identified with respect to the job.
- g. For microhardness, the hardness type and load.

7.14 Replication

Written procedures for replication shall cover cleaning, polishing, etching and examination. The procedures address avoidance of surface damage of dimensional discrepancies on the finished part. When required, the procedure ensures refinishing of replicated area to drawing requirements. The procedures are to be approved by the customer, if required. Prepared surfaces shall be free from scratches and artifacts, to permit proper evaluation.

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8. HARDNESS:

8.1 Hardness Test Types / Codes

M HARDNESS (General)
M1 HARDNESS (Brinell)
M2 HARDNESS (Rockwell)
M3 HARDNESS (Vickers)

COATINGS (AC7109/5)
HARDNESS (Rockwell)

8.1.1 Hardness testing by other sources (e.g., heat treat, plating, metal spray sources) may be approved as documented.

8.2 Test Methods and Procedures

8.2.1 The following apply unless otherwise specified:

ASTM E 10 Brinell Hardness
ASTM E 18 Rockwell Hardness
ASTM E 92 Vickers Hardness
ASTM E 140 Hardness Conversions

8.2.2 Written instructions shall cover the following testing variables.

- a. Number of impressions for each test value reported.
- b. Reading techniques for non-digital-readout testing
- c. In-process calibration procedures and recording.

8.2.3 Hardness testing equipment shall be designed to ensure that the force of the indenter is applied smoothly, and without impact (overload) forces being applied. The base of the equipment shall be constructed such that it is sufficiently rigid to react the test load, with no rocking or lateral movement of the specimen. The angular alignment of the indenter to the anvil shall be $90^\circ \pm 2^\circ$.

For Brinell hardness testing the system utilized shall eliminate, or dampen acceleration of the load as it approaches the intended level.

8.2.4 Rockwell hardness machines used for acceptance testing of material shall fall within the +/- tolerance for the standard test block. Correction factors to compensate for machine error shall not be used. If observed values exceed the tolerance of the test block, the equipment is adjusted and re-calibrated prior to use.

8.2.5 For Brinell, the measuring microscope shall have the accuracy to measure the diameter to 0.1mm and to estimate the diameter to 0.05mm. For Vickers, the accuracy to measure the diagonals shall be within +/- 0.0005mm or +/- 0.5%, whichever is larger. On manual systems two readings shall be taken at 90 degrees apart around the diameter.

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- 8.2.6 Hardness block identification shall consist of the following:
- An official mark on the test surface (traceable to its certification) or the thickness to an accuracy of ± 0.005 inches, to verify the original surface has not been ground. The surface shall be uniformly polished, and free from scratches that may affect current indentations.
 - The side of each block shall be marked with the serial number, the mean hardness value, tolerance (if given) and the supplier's identification.
- 8.2.7 Spacing of indentations on hardness test blocks shall be no less than $2\frac{1}{2}$ times the diagonal/diameter of the indentations from center to edge of indentation for Vickers/Brinell. For Rockwell the center to center spacing shall be no less than three (3) times the diameter of the indentation. The laboratory shall have a means of dispositioning test blocks which show evidence of spacing closer than allowed. Test blocks have indentations made only on the certified surface.
- 8.2.8 Whenever the anvil or indenter is changed, and before the machine is checked with a test block, the technician shall visually inspect the anvil and indenter. Preliminary indentations (may be on scrap stock) shall be made to ensure seating of the anvil and indenter.
Brinell and Vickers: 1 indentation minimum.
Rockwell: 2 indentations minimum.
- 8.3 Hardness Test Matrix
- Figure 1, or equivalent, in checklist AC7101/5, shall be completed to include all equipment which is used for acceptance testing of material. It shall include reference to applicable Round Robin programs.
- 8.4 Hardness Test Personnel
- Operators who read test machine dials or microscopically measure indentations shall be evaluated against other operators for consistency. Such evaluations may also be used to satisfy Round Robin requirements.

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8.5 Test Specimens

8.5.1 Optimum specimens have all of the following characteristics, and should be used for all tests which require both minimum and maximum limits.

a. All specimens

- Specimen center of gravity supported by standard anvil.
- Smooth test surface finish; maximum roughness.

Brinell	125 μin (3.20 μm)
Rockwell	63 μin (1.60 μm)
Rockwell superficial	32 μin (0.80 μm)
Vickers	16 μin (0.40 μm)
- Sufficient thickness so that testing is confined to the specimen (i.e., so that no bulge or other markings are present showing the effects of the test force on the specimen opposite the indentations). A general rule for thickness is 10 times the depth of the indentation for Brinell testing.
- No local protrusions on anvil-side surface.

b. Flat specimens

- Test surface and opposite surface machined or ground flat, with no local protrusions (such as mechanical marking) on the anvil-side surface; and parallel within 2.0°.
- Mounted specimens should be used for Rockwell superficial or Vickers hardness only.

c. Contoured specimens

- Cylindrical and spherical material (e.g., ball bearings, ground bar) with proper surface finish, tested in proper holding fixtures, and with appropriate correction factors.
- For Brinell hardness, the specimen radius shall be greater than 1 inch.

8.5.2 Non-Optimum specimens, such as large parts which require special fixturing or parts of irregular geometry, may be used only for tests which require minimum limits only. A minimum of two indentations shall be made and compared for consistency to +/- 3% to ensure that the specimen did not move.

8.6 Routine Calibration Checks

Routine periodic checks shall be made on standard test block(s) representing the hardness range(s)/loads/indenters to be tested. The results shall be recorded such that it is possible (if necessary) to retest those tests between the periodic checks.

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- 8.6.1 Periodic checks shall consist of 2 indentations for Brinell and Vickers and 3 indentations for Rockwell. The results must be within the tolerances on the test block.
- 8.6.2 Frequency: Check once each working shift during which the tester will be used. Equipment that can be changed by the operator (e.g., Rockwell testers with several loads/indenters): check prior to each test run, defined as testing of material using the same equipment set-up while the machine is under the control of the same operator.
- 8.6.3 If the periodic check reveals an out of tolerance condition, all tests since the last acceptable check shall be retested, unless the suspect population can be identified and segregated. For Vickers hardness, if the measurement in either direction differs more than 2% from the mean, the equipment shall be adjusted and re-verified.
- 8.6.4 The operator shall inspect the equipment prior to the periodic check, to ensure that there is no damage. This inspection shall include the anvil and indenter.
- 8.7 Hardness Re-Testing and Referee Tests
- 8.7.1 Hardness Re-Test: Because problems such as specimen wobbling may occur which are apparent only to the operator and only at the time of testing, operators may choose to disregard test impressions they suspect of being irregular. Provided two additional impressions are made, both of which give conforming values and whose variations are typical for the material and hardness procedure, the suspect test may be disregarded and need not be recorded in the log or on the certificate.
- 8.7.2 Hardness Referee Tests
- The laboratory shall have a procedure addressing Hardness Referee tests, which includes at least one of the following:
- Re-preparation of the specimen to improve surface finish, parallelism, and/or flatness.
 - When "alternate" test methods are permitted, use of a hardness method or scale that best represents the material being tested. For example:
 - Use of spherical indenter methods when material is work-hardenable.
 - Use of Brinell hardness on castings or other large-grain material.
 - Use of Brinell hardness when "Brinell or alternate" is specified.
 - Selection of load which produces maximum sensitivity of readings (higher loads for higher hardnesses).
 - Use of direct-reading digital equipment instead of equipment requiring human interpretation of readings.
 - Make three to five impressions. All readings should conform to specification; report the median value.

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8.8 Hardness Round Robin Testing

Round Robin testing shall be conducted in accordance with Appendix E.

8.9 Alternate Hardness Methods

Alternate hardness methods may be used when permitted by the material specification or otherwise accepted as a "non-standard practice" for the applicable application (ref. Table 1). Hardness conversions shall be reported per ASTM E 140. When Rockwell hardness is converted from Brinell, Brinell indentation diameters shall be measured to give an equivalent sensitivity of $\frac{1}{2}$ Rockwell number. Prior to use of ASTM E 140 or other conversions, a calibration report is required (primary versus equivalent method) for the following:

- Alloy groups not covered by ASTM E 140.
- Work-hardenable materials.
- Castings and other coarse-grain materials.
- Microhardness in lieu of Rockwell or Brinell, or vice versa.

8.10 Hardness Test Reports

Hardness test reports shall include the following:

- a. The Rockwell hardness number followed by the scale (i.e., HRC, HRB, HRA).
- b. The time of application of the total test force if greater than three (3) seconds (Rockwell).
- c. Vickers hardness number, followed by "HV".
- d. The test load used (Vickers).
- e. The loading time, if other than 10-15 seconds (Vickers).
- f. The test condition and test force application, if other than 3000 Kgf with a 10mm ball for 10-15 seconds (Brinell).

8.10.1 Rounding shall be performed in accordance with ASTM E 29.

8.11 Operating Parameters

The laboratory's procedures shall provide for control of operating parameters which can affect test data. The procedures shall address the attributes listed in this section.

8.11.1 Brinell Hardness Testing

- a. A 10 mm ball is used with the standard loads (500, 1000, 1500, 3000 kgf), unless required by the product specification.
- b. The load shall be selected to produce an indentation diameter in the range of 2.50 to 6.00 mm.
- c. Standard time of load retention is 10-15 s, unless specified by the product specification.

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8.11.1 Continued

- d. If equipment is to be used at conditions other than standard, periodic verifications shall be performed and documented.
- e. Equipment is checked against standard test blocks which are certified for the load, indenter and time to be used.

8.11.2 Rockwell Hardness Testing

For manually controlled equipment:

- a. The “minor” load shall be applied smoothly, to within ± 5 divisions of the set position.
- b. The major load shall be applied smoothly, and shall be removed in the following manner, dependent on material characteristics:
 - 1) For materials with some time-dependent plasticity, the load shall be removed within three (3) seconds.
 - 2) For materials with greater time-dependent plasticity, the load shall be removed three (3) seconds after the major load is applied.
 - 3) Dwell times greater than three (3) seconds shall be as specified in the product specification, and shall be documented.
- c. When equipment uses an analog dial for reading the value, operator training and inspection aids shall be used to avoid the introduction of parallax error.

8.11.3 Vickers Hardness Testing

- a. The test load shall be from 1 to 120 kgf and shall be reported.
- b. Material homogeneity shall be considered when selecting the test load.
- c. The test load application and removal is done smoothly, and shall be applied for a period of 10-15 seconds, unless otherwise required by the product specification. Non-standard durations shall be documented.

9. CORROSION:

9.1 Corrosion - Codes

Q CORROSION
Q₁ STRESS CORROSION

- 9.1.1 Corrosion testing by other sources (e.g., diffusion coating sources) may be approved and documented for the applicable process.

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9.2 Test Methods and Procedures

Methods and procedures for corrosion testing shall be as called out by the material specification. If none are specified, the applicable ASTM Method or Procedure from Volume 3.02 "Erosion and Wear; Metal Corrosion" shall be used. Reference specification: ASTM A 262 and ASTM B 117.

Written procedures shall be issued to identify the methods and procedures used, and to relate details of the test specification to the actual equipment and practices in that laboratory. Instructions shall incorporate lessons learned with regard to invalidation of testing.

9.3 Personnel

Personnel shall be trained in preparation and handling of specimens, in maintenance of equipment, and in corrosion theory as it applies to corrosion testing.

9.4 Test Specimens

Specimens shall be prepared and handled so as to preclude the introduction of foreign materials or non-representative stresses to surfaces. Specimens shall be cleaned Within one hour of testing. After cleaning, specimens will be handled with lint-free gloves. Handling of specimens shall be kept to a minimum to avoid contamination. If detergent is used for cleaning, the specimens are to be rinsed in de-ionized water, followed by an alcohol rinse, and dried. For stress corrosion specimens, the pre-test examination for mechanical cracking shall be performed using the same magnification, or higher, as will be used during and after the exposure.

9.4.1 Test Specimen Racks and Supports

The racks and supports used in specimen restraint are to be kept from contacting each other, or any material which may act as a wick. Placement shall be such that the atmosphere will circulate freely and evenly around the specimens. Racks and supports shall be constructed to provide sample suspension at an angle of 15-30 degrees from the vertical orientation, and parallel to the principle direction of the horizontal flow of the medium through the chamber. Specimens are to be arranged to prevent "pooling" of solutions in the chamber.

9.5 Salt Spray Testing

The cabinet shall conform to the requirements of ASTM B 117, and satisfy the following parameters:

- a. Constructed of materials that will not affect the corrosion properties of the specimens.
- b. Provide a means to prevent condensate from the chamber ceiling or fixturing falling onto test specimens.
- c. Ensure that solution which has contacted specimens does not return to the reservoir, unless specified otherwise.

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9.5 Continued

- d. Provide a compressed air supply that is cleaned of oil and dirt via 2 feet (610 mm) of suitable cleaning material, such as sheep's wool, excelsior, slag wool, activated alumina or a commercial cartridge. All cleaning media shall have an expiration indicator.
- e. Chamber venting shall minimize back pressure, and extreme air currents at the opening of the vent pipe are to be avoided.
- f. The solution reservoir level is to be automatically maintained, to ensure consistent fog delivery, or constant humidity levels.

9.5.1 Test Chamber Conditions

The following conditions are applicable to all test chambers:

- a. Distilled or de-ionized water is used.
- b. Chamber temperature is recorded while the chamber is closed.
- c. Immersion heaters are not used in the chamber, nor in the reservoir.
- d. The chamber temperature is maintained to avoid overheating which causes evaporation of condensation, followed by condensation of that moisture on other specimens.
- e. The test chamber is kept sufficiently clean to avoid introduction of corrosive agents (e.g., salt concentrations).
- f. The temperature within the exposure zone shall be $95^{\circ}\text{F} \pm 2^{\circ}/-3^{\circ}\text{F}$.
- g. Atomization nozzles shall be made from inert material.
- h. Compressed air supply pressure shall be between 10 and 25 psi.
- i. Two clean fog collectors are required within the exposure zone, so that no drops of solution from the test specimen or other source are collected.
- j. The quantity of fog shall be such that for each 80 cm^2 of horizontal collecting area, a collector will collect between 1.0-2.0 ml of solution per hour, based on an average of at least 16 hours.

9.5.2 Salt Solution

Salt solutions shall conform to the following parameters, unless otherwise required by contract:

- a. The solution shall be $5\% \pm 1\%$ salt, by weight, in 95 parts distilled or de-ionized water.
- b. Only reagent grade salt may be used (shall be free from copper and nickel, and contain less than 0.1% of Sodium Iodine). Total other impurities are not greater than 0.3%.
- c. The pH of the solution, atomized at 95°F , is in the range of 6.5-7.2, and shall be measured electromagnetically, colorimetrically or with short range litmus paper. Daily checks are required.
- d. The pH of the solution is adjusted using only reagent grade Hydrochloric Acid or Sodium Hydroxide.
- e. The salt concentration is checked using a hydrometer, or equivalent, that has been tested in accordance with ASTM E 126.

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9.5.3 Specimen Examination

The specimens shall be examined immediately after exposure. If necessary, cleaning is to be done in clean, warm running water, with a temperature not exceeding 100°F to remove salt deposits.

9.6 Recorded Data

The following data shall be recorded in the test record, if not inherent in the procedure or operating specification:

- a. The type of salt water used in the solution.
- b. All temperature readings within the exposure zone of the test chamber.
- c. All corrective maintenance performed on the chamber.
- d. All types of specimens, dimensions of specimens and number or description of specimens.
- e. Method of specimen cleaning before and after exposure.
- f. Method of supporting or suspending the specimens.
- g. Method of protecting all cut edges.
- h. All interruptions in the exposure of the test specimens, including cause, length and time.
- i. Total exposure time.

9.6.1 Reported Data

The following information is to be reported on the test certificate:

- a. The temperature within the exposure zone, if it is other than 95°F, +2/-3°F.
- b. Specimen type, description, quantity and dimensions.
- c. Total exposure time.
- d. All results obtained during post-exposure examination.

9.7 Intergranular Attack in Austenitic Stainless Steel (ASTM A 262)

The laboratory shall have the capability of performing the examinations in accordance with the practices defined in ASTM A 262 (A,B,C,D,E,F), as applicable to their facility.

9.7.1 General Equipment/Operations

The following is required for performance of IGA testing:

- a. Heat treating capability for sensitization of specimens in accordance with AC7101/9.
- b. Machining capability to prepare the appropriate specimen configuration.
- c. Heating device to maintain the solution at a constant boiling level for the required exposure time.
- d. Analytical balance with an accuracy of 0.001 gram.

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9.7.2 Practice A Requirements and Reporting

- a. The laboratory shall have metallographic capability, including electrolytic etching, in accordance with AC7101/4.
- b. The certificate shall evidence the etched structure found, and the acceptance/rejection of the screening process.

9.7.3 Practice B Requirements and Reportings

The laboratory shall have the following equipment:

- a. Four bulb Allihn or Soxhlet condenser, or equivalent.
- b. Glass cradle, Erlenmeyer flask and boiling chips.
- c. Sulfuric acid of the appropriate concentration.
- d. The appropriate grade of Ferric Chloride.

9.7.3.1 The following items are to be evidenced on the certificate:

- a. Condition of the specimen (as received or sensitized, time and temperature of thermal treatment).
- b. Weight of specimen prior to exposure.
- c. Exposure time.
- d. Weight of specimen after exposure.
- e. Results of the exposure in inches/mils per month/year.

9.7.4 Practice C Requirements and Reportings

The laboratory shall have the following equipment:

- a. 1000ml Erlenmeyer flask, with a coldfinger type condenser or equivalent.
- b. Glass hooks, stirrups or cradles.
- c. Nitric Acid of the appropriate concentration.

9.7.4.1 The following items are to be evidenced on the certificate:

- a. Condition of the specimen (as received or sensitized, time and temperature of thermal treatment).
- b. Weight of specimen prior to exposure.
- c. Exposure time.
- d. Weight of specimen after exposure.
- e. Results of the exposure in inches/mils per month/year for each period, as well as the average for the number of exposures.

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9.7.5 Practice D Requirements and Reportings

The laboratory shall have the following equipment:

- a. PVC test cylinders.
- b. TFE fluorocarbon holding devices.
- c. Constant temperature water bath.
- d. Nitric and Hydrofluoric Acid of the appropriate concentrations.

9.7.5.1 The following items are to be evidenced on the certificate:

- a. Condition of the specimen (as received or sensitized, time and temperature of thermal treatment).
- b. Weight of specimen prior to exposure.
- c. Exposure time.
- d. Weight of specimen after exposure.
- e. Results of the exposure showing the corrosion rate ratio.

9.7.6 Practice E Requirements and Reportings

The laboratory shall have the following equipment:

- a. 1000ml Erlenmeyer flask, or four bulb Allihn condenser, or equivalent.
- b. Electrolytic grade copper shot or grindings.
- c. Facilities to perform the bend test in accordance with ASTM E 290 (Ref. AC7101/3, Code XN).
- d. Sulfuric acid of the appropriate concentration.
- e. Copper Sulfate.
- f. A 5X-20X magnification device.

9.7.6.1 The following items are to be evidenced on the certificate:

- a. Condition of the specimen (as received or sensitized, time and temperature of thermal treatment).
- b. Angle of bend, if other than 180 degrees is used.
- c. Observations after bending (presence/absence of fissures/cracks, etc.).

9.7.7 Practice F Requirements and Reportings

The laboratory shall have the following equipment:

- a. 1000ml Erlenmeyer flask, or four bulb Allihn condenser, or equivalent.
- b. Glass cradle.
- c. Electrolytic grade copper shot or grindings.
- d. Sulfuric Acid of the appropriate concentration.
- e. Copper Sulfate.

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- 9.7.7.1 The following items are to be evidenced on the certificate:
- Condition of the specimen (as received or sensitized, time and temperature of thermal treatment).
 - Weight of specimen prior to exposure.
 - Exposure time.
 - Weight of specimen after exposure.
 - Results of exposure in inches/mils per month/year.

9.8 Alternate Immersion Stress Corrosion

Alternate Immersion Stress Corrosion shall be conducted in accordance with ASTM G 44.

The test apparatus shall be constructed of materials that will not affect the corrosion of the test specimens, and do not cause galvanic corrosion. Safeguards are to be in place to prevent condensation from other specimens or fixtures to deposit on the test specimens. Back-pressure within the set-up is to be kept to a minimum, and the air temperature maintained at $80^{\circ}\text{F} \pm 2^{\circ}\text{F}$, and relative humidity at $45\% \pm 10\%$ for the entire test cycle.

The test apparatus shall be equipped with an alternate immersion mechanism, that provides for 10 minutes of immersion, and 50 minutes of drying time. If a Ferris wheel apparatus is used, temperature of the test chamber shall be maintained within tolerance at all height levels. There shall be a mild circulation of air around all specimens, forced blasts and heated are to be avoided. The rate of immersion shall not exceed 2 minutes from initial contact to total immersion.

Pockets or recesses in the test specimens shall be such that they are able to drain and dry within 50 minutes. Only like alloy families shall be exposed simultaneously in the same solution. All specimens are to be protected from exposure to fumes, or airborne contamination.

9.8.1 Salt Solution

The salt solution shall be 3.5% by weight, in 96.5 parts distilled or de-ionized water, with chlorides of less than 200 ppm. The salt solids shall be free from Nickel, Copper and other impurities. The pH of the solution, when measured weekly at $75^{\circ}\text{F} \pm 5^{\circ}\text{F}$, is in the range of 6.5-7.2, and shall be measured electrometrically, colorimetrically, or with litmus paper. Adjustments are made using only analytical reagent grade materials.

The concentration of salt shall be checked using a hydrometer that has been tested in accordance with ASTM E 126. The solution shall be replaced at least once a week. Evaporation losses are to be replaced with water of the required purity. When changing the salt solution, all parts of the apparatus which contact the solution require cleaning by flushing with distilled, or de-ionized water. The ratio of salt solution to specimen area shall be at least 200 ml/in^2 (32 ml/cm^2).

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9.8.2 Recorded Data

The following data shall be recorded in the test record, if not inherent in the procedure or operating specification:

- a. Alloy, temper and section thickness of material.
- b. Size and orientation of test specimen, relative to the as-fabricated material.
- c. Method of stressing and level.
- d. Test duration.
- e. Any interruptions in the test procedure.
- f. The pH level.
- g. Concentration of the salt solution.
- h. Fluctuations in temperature outside of the prescribed range.

9.8.3 Reported Data

The following information is to be reported on the test certificate:

- a. Alloy, temper and section thickness of material.
- b. Size and orientation of test specimen, relative to the as-fabricated material.
- c. Stress level.
- d. Test duration.
- e. Any interruptions in the test procedure.
- f. Results from any metallographic examinations.

9.9 Bent Beam, C-Ring, Direct Tension and U-Bend Stress Corrosion

9.9.1 Sample Preparation (applies to all Section 9.9 tests)

Orientation of test specimens, as cut from parent material, shall be defined. Sample preparation operations are done in stages, so that the final surface cut leaves a finish of 30 μ inches (762 μ m) or better. Excessive heating during preparation, that may induce undesired residual stresses and metallurgical/chemical changes, is to be avoided.

Test specimens shall be identified on both ends, in such a way as not to interfere with stressing or corroding of the specimens, and is affixed in a manner that will not be obliterated by the corrosion. Solutions used to degrease the test specimens are to be non-reactive to the material and shall be residue-free. No chemical or electrochemical treatments, which produce hydrogen, or react with hydrogen to form hydrides, are permitted, as their use may induce hydrogen embrittlement in some materials.

Precautions shall be taken after final preparation, to prevent contamination or marring of the test specimen surface. The specimens are to be introduced into the environment as soon as possible after degreasing, stressing and inspection. Inspections for mechanical cracking prior to testing are to be performed at the same or higher magnification as used during the test.

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9.9.1 Continued

Periodic inspections performed during testing shall ensure that the stress is not grossly relieved during testing due to crack propagation or test fixture relaxation. If the inspections require that the specimens be removed, they shall be returned to the test chamber uncontaminated by any non-test constituent. Care must be used not to change the stress level on the test specimen. The time outside the test chamber shall be kept to a minimum, and is not to be included in the total exposure time. Cleaned specimens are not to be returned to the test environment unless it is the intention of the test to evaluate this variable.

Galvanic reactions between the specimen, the stressing bolt and the exposure rack are to be avoided. Specimens shall be supported in such a manner that nothing but the corrosive medium comes into contact with them.

9.9.2 Bent Beam Testing (ASTM G 39) -- Method Particulars

If alternate immersion, salt spray, humidity or any other method of testing is used in conjunction with bent beam testing, specifications for the test environment are in accordance with the appropriate procedure. Bent beam tests are designed for testing at stress levels below the elastic limit, while avoiding tests performed in the plastic range.

Specimen holders shall be made of a material that will withstand the effects of the test environment, without deterioration or deformation, and be sufficiently rigid to retain the applied stress on the specimen. If tests are performed in an electrolyte, galvanic reaction between the specimen and the holder is to be avoided. When an electrolyte is used and crevice corrosion may occur at contact points, hydrophobic fillers shall be packed into these areas, or specially designed holders are to be utilized.

Specimen preparation must conform to ASTM G 39. Specimens shall be machined into flat strips of uniform rectangular cross section and thickness. If stenciling is used for identification, it shall be performed prior to any hardening heat treatments. Unless specified otherwise, specimens shall be excised so as to retain the original material surface. When surface material removal is required, it shall be performed in staged operations, so that the final surface finish is at least 30 $\mu\text{in.}$ (0.7 μm) or better, with at least 0.01 inch (0.25 mm) of material removal from each side and the edges of the specimen. If cold working is present, these edges are ground or machined to remove the effect. The specimen surface condition is recorded and reported.

9.9.3 C-Ring Stress Corrosion (ASTM G 38) -- Method Particulars

If alternate immersion, salt spray, humidity or any other method of testing is used in conjunction with C-Ring testing, specifications for the test environment are in accordance with the appropriate procedure.

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9.9.3 Continued

Specimen preparation must conform to ASTM G 38. Specimens shall be cut from the original stock so that the direction of principal stress is in the direction of minimum resistance to stress corrosion cracking. Operations such as lapping or mechanical polishing, which produce flow of the metal are prohibited. If strain gages are used to measure strain, they are to be placed in the center of the arc to detect maximum strain. A correction for the displacement of the gage from the surface of the ring is permitted.

The strain gage adhesive shall be completely removed from the specimen prior to testing.

If etchants are used, they shall be of a type that will not induce hydrogen embrittlement in materials that are susceptible, and do not selectively attack constituents in the metal. Only etchants that do not leave an undesirable residue on the sample surface are permitted. All surface preparation, except for final degreasing when required, shall be completed prior to stressing of the specimen.

The date and time of the first evidence of cracking is to be noted while the specimen is kept in the test environment. Exposure shall be discontinued, and a metallographic examination of the cross section taken to confirm cracking.

9.9.4 Direct Tension Stress Corrosion (ASTM G 49) -- Method Particulars

If alternate immersion, salt spray, humidity or any other method of testing is used in conjunction with Direct Tension testing, specifications for the test environment are in accordance with the appropriate procedure.

Specimen preparation must conform to ASTM E 8, whenever possible. If possible, the specimens are to be stressed while already in the corrosive environment. Care shall be exercised in the construction of stressing frames to avoid bending stresses. An alignment check shall be performed and the results maintained on file.

9.9.5 U-Bend Stress Corrosion (ASTM G 30) -- Method Particulars

If alternate immersion, salt spray, humidity or any other method of testing is used in conjunction with U-Bend testing, specifications for the test environment are in accordance with the appropriate procedure.

Specimen preparation methods shall be consistent, unless the intent of the test is to evaluate this variable. If comparison studies are conducted, specimen dimensions are to be kept constant, especially the thickness to bend radius ratio. All thermal treatments are to be performed before final preparation, and the edges of specimens shall have the same finish as the specimen face.

When final surface preparation requires chemical dissolution, the solution used is not permitted to induce Hydrogen embrittlement, selectively attack constituents nor leave any residue on the specimen surface.

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9.9.5 Continued

When specimen preparation requires no metal removal, the specimen edges are mechanically removed.

The single stage, or two-stage method of stressing, in accordance with ASTM G 30, is required to stress the specimens. When the two-stage method is used, care shall be taken to prevent pre-straining greater than the final test strain, and to avoid "spring back" of the legs, after achieving final plastic strain.

The apparatus used to maintain the required stress level shall be insulated from the test specimen, to prevent any galvanic reaction.

9.9.6 Recorded Data

The following data shall be recorded in the test record, if not inherent in the procedure or operating specification:

- a. Time and date of each observation, initiation of failure and failure.
- b. Specimen dimension and surface preparation.
- c. Magnitude of the applied stress.
- d. Specimen orientation and identification.
- e. Time outside the test environment used for examination.
- f. Composition of the test lot.
- g. Details concerning the material.
- h. Test medium.
- i. Remarks about the size and appearance of cracks.

9.9.7 Reported Data

The following information is to be reported on the test certificate:

- a. Specimen dimension and surface preparation.
- b. Method of stressing each specimen.
- c. Time cracking becomes visible, if applicable.
- d. Magnification used to view the cracks and defects.
- e. The percentage of specimens cracked, if multiple specimens are tested.
- f. Pitting or surface defects.
- g. Explanation of any interruptions in testing.
- h. Total exposure time.
- i. Test medium.
- j. Failure criterion.
- k. Composition, temper and alloy of the test lot.

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9.10 Exfoliation Corrosion Susceptibility in 2XXX & 7XXX Aluminum Alloys

Exfoliation Corrosion testing shall be conducted in accordance with ASTM G34.

9.10.1 Test Chamber

The test chamber, and rods or racks which hold the specimens, shall be constructed of materials that will not affect the corrosion of the specimens, and shall have a loose fitting lid to minimize evaporation.

9.10.2 Test Solution

The solution is to consist of the following reagent-grade constituents:

- a. Sodium Chloride (NaCl), 4.0M
- b. Potassium Nitrate (KNO₃), 0.5M
- c. Nitric Acid (HNO₃), 0.1M

Distilled or de-ionized water that conforms to ASTM D 1193, Type IV, shall be used for the solution. The temperature shall be maintained at 77°F ±5°F.

9.10.3 Specimen Preparation

Care shall be taken when excising specimen blanks from extrusions or forgings, to avoid locations beneath flanges, ribs and other areas where the grain structure may vary. The edges of sheared or blanked specimens are to be machined to remove any cold worked material. Degreasing is done with a suitable solution that leaves no residue, and will not affect the test. Non-test areas of the specimen are to be masked to minimize corrosion in these areas, and the coating should have good adherence to avoid crevice corrosion. Protective coatings shall be free of leachable ions, or protective oils which may influence corrosion of the test surface.

9.10.4 Exposure

The same solution shall be used for the duration of the exposure, and be of sufficient volume to provide a volume-to-metal ratio of 65 to 200 ml/in² (10 to 30 ml/cm²). The specimen surface areas shall face upward to prevent loss of exfoliated material. The exposure of alloys containing less than 0.25% Copper, along with alloys containing greater than 0.25% Copper, is not permitted.

A control specimen, of known resistance, is to be used regularly to standardize the test conditions.

Specimens shall be inspected periodically for exfoliation, in accordance with ASTM G 34, Section 12. The maximum exposure period for 2XXX alloys is 96 hours, and for 7XXX alloys is 48 hours.

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9.10.4 Continued

The specimens are to be inspected and rated immediately after discontinuation of exposure, while they are still wet, or moist. Loose products of exfoliation shall be taken into account. If desired, specimens may be cleaned in Nitric Acid after inspection and rating. If added exposure time produces more corrosion, but no evidence of delamination, metallographic examinations are to be conducted to determine whether the initial effect is truly exfoliation. When exfoliation occurs in isolated areas, the worst localized condition is to be rated, and the final rating of a specimen is to be determined by the poorest classification observed during the exposure. When it is difficult to classify a specimen, it shall be placed in a category of greater susceptibility.

Specimens are to be classified by visual ratings in accordance with ASTM G 34, Section 12. The laboratory shall have glossy prints of exfoliation examples, which shall be used for visual rating comparisons. The ratings are classified as follows:

- a. "N" (No appreciable attack) Surface is discolored or etched, and shows no evidence of pitting or exfoliation.
- b. "P" (Pitting) Specimen has discrete pits, sometimes with a tendency for undermining and slight lifting of metal at the edge of the pit.
- c. "EA" (Superficial) Specimen shows exfoliation, with tiny blisters, thin slivers, flakes or powder, and slight separation of metal.
- d. "EB" (Moderate) Specimen shows exfoliation with notable layering and penetration into the metal.
- e. "EC" (Severe) Specimen shows exfoliation with penetration to a considerable depth into the metal.
- f. "ED" (Very Severe) Specimen shows exfoliation with much greater penetration into the metal than "EC".

9.10.5 Recorded Data

The following data shall be recorded in the test record, if not inherent in the procedure or operating specification.

- a. Size and type of each specimen.
- b. Alloy, temper, mill product and material thickness.
- c. Any deviations from ASTM G 34.
- d. Method of specimen edge preparation.
- e. Volume to surface ratio.

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9.10.6 Reported Data

The following information is to be reported on the test certificate:

- a. Size and type of each specimen.
- b. Alloy, temper, mill product and material thickness.
- c. Any deviations from ASTM G 34.
- d. Rating of each specimen in accordance with ASTM G34, Section 12.
- e. Method of specimen edge preparation.
- f. Volume to surface ratio.

9.11 Visual Standards

When the laboratory is responsible for acceptance of test results (rather than simply performing the tests), visual or photographic standards shall be maintained for acceptance to specifications which permit acceptance of some degree of visible corrosion.

9.12 Continuation of Testing (Corrosion/Stress Corrosion):

If nonconformance to required life is noted in one specimen among multiple specimens from the same set, it is recommended that testing of remaining specimens be continued beyond 1.5 times the required life or until failure, whichever time is less. For proposed acceptance of unexplained nonconformances, a set should contain at least three acceptable tests for each nonconformance. All results should be reported on the nonconformance document.

10. MECHANICAL TESTING SPECIMEN PREPARATION:

10.1 Specimen Preparation Codes

- Z STANDARD PREPARATION
- Z1 LOW STRESS GRINDING
- Z2 LOW STRESS GRINDING AND POLISHING
- Z3 CAST SPECIMENS
- Z4 SPECIAL PURPOSE SPECIMENS

Codes "Z1 and Z2" apply to Test Codes "Y, XH", and to any other test specimen for which the material specification requires low stress grinding (LSG). Other requirements of this section apply to preparation of all mechanical test specimens.

10.2 Preparation Procedures:

Written procedures shall be maintained and followed which establish specific values for each combination of specimen and material, for each process variable (e.g., speed, feed, grinding wheel type); these may include reasonable tolerances to recognize limits of process controllability and material response. A 100% verification or SPC is required for all gage section dimensions or other dimensions which might affect testing axiality or stress concentration.

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10.3 Technical Objective and Requirements:

Ideally, specimen preparation techniques eliminate any effects of preparation on the properties resulting from the test. Factors that must be considered include:

- Distortion of specimen dimensions.
- Physical damage (e.g., cracks, tears, scratches).
- Residual stresses (tensile or compressive).
- Metallurgical damage (e.g., local overheating, melting, decarburization, alloy depletion).

10.3.1 Straightening: Mechanical straightening of specimens prior to testing is not permitted. Exception: Castings and as-cast test bars which permit straightening followed by solution treat and age.

10.3.2 Blanking: Blanking may be done by machining, grinding, Electrical Discharge Machining (EDM), pressing (flat specimens), or other methods which do not distort the material or produce surface effects deeper than would be removed by final finishing. A minimum of 0.010 inch (0.25 mm) shall remain on blanked surfaces for final finishing operations. For sheet specimens, as-finished sheet surfaces should remain intact.

10.3.3 Special Purpose Specimens: For testing of weld and braze joints, tubes, miniature bars, and other material conditions which are not suitable for standard flat or cylindrical specimens, blanking and finishing operations shall be covered by written procedures.

10.3.4 Inspection: Visual inspection (20X) of finished specimens is required for Code Z2 and is recommended for other specimens.

10.3.5 Test specimen conditions which may affect the results shall be recorded prior to the test.

10.4 Notch Machining

The surface finish of notches shall conform to customer requirements, or to parameters established by the laboratory. The notch shall be free from scratches or nicks which may cause premature fracture. Notch dimensions shall be provided with the specimen drawing.

10.4.1 Inspection of Specimens

Each specimen shall be inspected prior to testing, except for the notch. The inspection shall be at 1X minimum. Magnifications other than 1X shall be noted in the test record. If inspection is achieved by NDT methods, this must be noted in the test record.

10.4.2 Test Specimens shall be inspected for the following attributes:

10.4.2.1 The surface has a uniform surface texture, free from machining irregularities such as undercuts or high spots.

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10.4.2.2 Critical dimensions are inspected using one of the following plans:

- a. 100% of all machined dimensions are inspected and recorded.
- b. 100% inspection of all reduced section dimensions; sample inspection of other dimensions. Measurements are to be recorded.

10.4.2.3 Sample inspection of specific dimensions conforming to the parameters listed below, with the measurements being recorded:

- a. Sampling plans, in the laboratory, are based on documented procedures.
- b. Sampling plans of outside source are approved by the laboratory.
- c. When sampling plans are used, Statistical Process Control is used for data analysis and control.

10.4.3 Notched sample inspection shall be performed at 10X magnification minimum, with the following characteristics documented:

- a. Notch radius
- b. Notch angle
- c. Diameter/dimension at root of the notch

10.5 Grinding of "Z2" Specimens

Final grinding of smooth section specimens shall be performed in accordance with values specified by the customer/specification. As a minimum, the lubricant, surface speed (sfm) and down feed (pass per revolution) shall be specified.

10.6 Final Polishing of "Z2" Specimens

Final polishing activities shall conform to customer/specification requirements, and shall encompass the following attributes:

- a. For titanium alloys, silicon carbide abrasives are required; for all other alloys, aluminum oxide abrasives are required.
- b. Round bars shall be polished with automated equipment, unless specified by the customer. Automated equipment shall ensure that contact between the specimen and a specific area on the abrasive is one (1) stroke on the paper in the axial direction.
- c. Paper is backed with a rubber pad, 3/16 inch minimum thickness, and an applied force of 6 pounds (2.7 kg) maximum.
- d. Circumferential grinding marks are to be removed using 180-240 grit abrasive prior to proceeding with the finer grits.
- e. Subsequent grit paper polishing is continued until all evidence of previous grit polishing has been removed.
- f. Surfaces shall not be polished to a "mirror" finish.

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- 10.6.1 The polishing process aims for material removal in specific increments as listed below, as required to meet final dimensions:
- a. 180-240 grit = 0.0005 inch (12.7 μm)
 - b. 400 grit = 0.0003 inch (7.6 μm)
 - c. 600 grit = 0.0002 inch (5.1 μm)
 - d. The final polished surface finish shall be 8-12 Ra $\mu\text{in.}$ (0.2-.03 μm) or as specified by the customer.
- 10.6.2 Code "Z2" specimens shall be inspected at 20X magnification, and shall exhibit no cracks or transverse indications.
- 10.7 Round Robin, Code "Z2" Low Cycle Fatigue (LCF) Specimen Preparation
- Laboratory Accreditation shall include participation in a NADCAP sponsored Round Robin program; see Appendix E.
- 10.8 Residual Stresses (Code Z1 and Z2 specimens only)
- It is recommended that residual stresses be determined initially for each preparation procedure: thereafter, an audit plan should provide for evaluation of a minimum of one procedure per year, with all procedures being evaluated in the shortest possible time. Residual stresses shall be measured by X-Ray Diffraction, and be within the range of 20-85 ksi (140-590 MPA) compression, or as specified by the customer for the specific process.
- Note: At least 5 measurements (1 surface and 4 below) are needed to obtain a valid surface measurement.
11. DIFFERENTIAL THERMAL ANALYSIS (DTA):
- 11.1 Test Types - Codes (DTA)
- XJ DIFFERENTIAL THERMAL ANALYSIS (DTA)
- 11.2 DTA Test Methods and Procedures
- Methods and procedures for DTA shall be developed by the laboratory and validated against metallographic methods. Procedures shall include photomicrographs which define the metallurgical transformations to be evaluated.
- 11.2.1 Written procedures shall identify the specific equipment, parameters and procedures used.
- 11.2.2 Procedures shall be consistent with validation studies for each alloy tested.

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11.2.3 Procedures shall include the following:

- a. Gas type and flow
- b. Heating rate
- c. Cooling rate

11.2.4 When applicable, procedures or standards provided by the material producer shall be used.

11.2.5 Procedures for titanium alloys shall list the applicable heating rates for each specific alloy.

11.2.6 Specimen Re-Cycling: Each specimen shall be subjected to heating and cooling through the phase transformation cycle only once, unless otherwise specified or permitted by the customer's testing instructions.

11.2.7 Procedures or standards from the material producer or material customer shall be used whenever available.

12. HEAT TREATING.

12.1 Test Types - Codes

XG HEAT TREATING OF SPECIMENS

COATINGS (AC7109/5)

HEAT TREATING OF SPECIMENS

Bonding Of Specimens (Adhesives)

This code applies to heat treatment other than production heat treatment of the material represented. For example: Heat treatment of specimens, partially machined blanks, separated samples (test rings, prolongs); sample parts heat treated in the laboratory rather than in production facilities; specimen material aged for capability properties when material is to be supplied in the solution treated condition. If the laboratory heat treats production parts, they shall also be qualified to AS7102.

12.2 Personnel

Personnel shall be trained in handling of specimens and use of equipment commensurate with applicable requirements, including furnace loading and specimen quenching.

12.2.1 Material Identification and Control

Test material shall be identified by a unique number, and handled in a manner which maintains traceability to this number throughout all processing. The information required to be traceable to this number shall be specified in Appendix A of this standard.

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12.3 Furnaces

Furnaces shall be surveyed to the requirements of AMS 2750 Class 1 or by an equivalent procedure approved by the Purchaser. For captive laboratories of material sources, surveys may be conducted per the applicable alloy heat treat specification (e.g., MIL-H-6875 for steels). Working zones shall be established by furnace survey and defined by a procedure. The zones shall be posted near the furnace. Minimum frequency of temperature uniformity surveys shall be per AMS 2750, except that minimum frequency shall be initially (new equipment) with annual verification.

12.3.1 Furnace type shall be in compliance with customer/contract/specification requirements, and shall be equipped with time/temperature recorders.

12.4 Time and Temperature

Heat treat times, temperatures and ramp rates shall be as specified by the customer, or applicable specification. Temperature shall be set to achieve the nominal specified value as closely as possible. Furnaces shall be equipped with over-temperature sensors. Metal temperature tolerances are intended only to limit maximum furnace tolerances. Times and temperatures shall be recorded on the certificate of test. If time tolerances are not given, specified time is minimum, minus zero, plus 10%. Time is measured from the time that the thickest section of the metal reaches the required temperature; the laboratory shall have a written rationale for determining this time (e.g., for light loads of a few well-spaced specimen blanks, time cycle might begin when the furnace recovers to temperature).

12.5 Quench Method

When options are permitted in quench rate (e.g., specification states "air, oil, or water quench"), the slowest method shall be used unless otherwise specified by the customer. Specimen racking, when required, shall provide for proper quenching and minimum distortion/damage to specimens. Facility shall be arranged to provide for expedient quenching.

RATIONALE: Slower quench rates tend to produce lower tensile strength in Quench & Temper and Solution & Age alloys. For example, properties of water quenched specimen material may not adequately represent oil or air quenched parts. (In most cases, specimen material has less effective thickness -- hence a naturally faster cooling rate than the parts represented even when the same type quench is used.)

12.6 Specimen Condition

Unless otherwise specified, specimens shall be heat treated in the blanked or rough machined condition.

12.6.1 Heat Treatment of Finished Specimens: If heat treating of finished specimens is specified, specimens shall be inspected for evidence of distortion prior to testing.

12.6.2 Written procedures shall describe in detail instructions for precleaning, pretreating and postcleaning.

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12.7 Certificate of Test

The following items shall be described:

- a. Heat treat times and temperatures.
NOTE: Test records shall document metal time at temperature (heaviest section), including tolerances, and temperature tolerances.
- b. Quench methods.
- c. If heat treating was performed on finished specimens, document this fact and describe the atmosphere.

12.7.1 All heat treat cycles are to be logged against a unique number.

12.8 Distortion Control

Heating and cooling shall be controlled to minimize stresses that could cause distortion or dimensional instability during machining or testing. Mechanical straightening of specimens after heat treatment is not permitted.

12.9 Calibrations

Calibration of thermocouples and pyrometry equipment shall conform to the requirements of AMS 2750 and shall be documented.

12.10 Furnace Atmosphere

Furnace atmosphere shall be controlled in accordance with customer requirement, or applicable specification.

13. X-RAY DIFFRACTION EVALUATION (XRD):

13.1 Test Types, Codes, Methods and Procedures

Codes are listed below. The test specifications and standards are listed for information only. Specific methods are to be specified by the customer; or, if not specified, by the laboratory.

<u>Code</u>	<u>Test Type</u>	<u>Test Spec/Standard</u>
XB	Residual Stress Measurement	ASTM E 915/SAE J 784
XC	X-Ray Crystallography	None-/ASTM E 82
XR	Retained Austenite Measurement	ASTM E 975/SAE SP-453

The laboratory shall have the appropriate support equipment as required for this discipline. The equipment shall be maintained in good working order.

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13.2 Personnel and Training

Laboratory management shall assign responsibility for review and approval of test procedures, test results and authorization of retesting. Operators are to be trained to recognize proper versus improper operation of the equipment. Training should include examples of past problems.

Degreed engineers, or equivalent, are required for the following functions:

- a. Review and approval of data to be certified.
- b. Interpretation of raw data from equipment with non-numerical readouts.
- c. Authorization of retesting for nonconforming values.
- d. Preparation or approval of detailed internal testing procedures.

13.3 Specimen Preparation

Written procedures shall be established which preclude the introduction of foreign materials, altering of physical form or phases present, or altering or abrading the surface prior to measurement. For retained austenite measurements, the amount of surface material removed shall be in accordance with the testing specification.

13.4 Alignment Verification

Alignment of X-Ray Diffraction equipment shall be checked regularly, and refreshed as necessary. Alignment shall be documented, and alignment/verification methods and records are to be defined in an internal procedure. Standards shall be properly documented, and referenced to a governing body (NIST, Round Robin, etc.).

The laboratory shall have a documented procedure to maintain and align the system, and shall have a reference specimen to serve as an alignment check standard. This specimen must produce an identical pattern to the reference original, prior to running unknown specimens.

13.5 X-Ray Diffraction Residual Stress Measurement

Measurement Method

Initial measurements shall be made by the sine-squared-psi technique. The linearity of the lattice spacing shall be ± 3 ksi for samples under consideration.

In general, the psi angles shall be between 0° and 45° . When this is not possible, the difference in the psi angle must be greater than 30° .

Data shall be collected to determine the background intensity, and slope, for the purpose of correcting the diffraction peak intensity measurements. The diffracted intensity shall be determined for at least five (5) points, spaced at even increments across the diffraction peak, or by step scanning. The time shall be recorded at each point, to assure collection of well-defined peaks.

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13.5 Continued

The operator shall be familiar with the potential sources of error associated with grain size, stacking faults, twinning and texture of the specimens.

The residual stress shall be calculated directly from the strain in the crystal lattice, rather than diffraction peak position shift. The laboratory must have a method of assuring that the incident beam height and width do not deviate more than 0.002 inches from a flat surface.

13.5.1 Subsurface Residual Stress Measurements

Material shall be removed by electropolishing or acid etching of thin layers. If a combination of machining or grinding and electropolishing is used, a minimum of 0.008 inches of material shall be removed by electropolishing, to ensure complete removal of residual stresses induced by the machining/grinding operation.

A stop-off coating shall be used to protect all but the measurement area of the specimen. Etching and pitting shall be controlled by the use of appropriate acid solution and current density, and the electropolishing operation documented in a procedure. Caution shall be exercised to avoid abrading the surface prior to measurement.

Radiation Penetration Correction

X-Ray Diffraction data obtained as a function of depth shall be corrected for the exponential weighting of the measured lattice spacing caused by the penetration of the X-Ray beam into the subsurface lattice spacing gradient. The gradient correction shall be performed independently for each of the psi angles employed for measurement, before re-calculation of the gradient corrected stress.

Stress Relaxation Correction

When it is necessary to section a specimen, to expose the stress measurement site, or to reduce the overall size of a specimen to manageable proportions, the sectioning strain relaxation shall be monitored at the stress measurements site, using a three-grid strain gage rosette. A Biaxial rosette may be used, if the directions of principal stress relaxation are known.

XRD residual stress measurements shall be corrected using the sectioning stress relaxation data.

The determination of residual stress by XRD, and strain gaging operation shall be clearly defined in a written procedure, and abrading of the strain gage location is not permitted prior to measurement. When material is to be removed by electropolishing to determine the subsurface residual stress distribution, the residual stress distribution is corrected for stress relaxation which occurs as a result of layer removal, in accordance with Moore and Evans (SAE Transactions, Vol. 66, 1958).

All raw data shall be stored for calculation at a later time.