Polyacrylonitrile-Based Carbon Fiber Process Control Document (PCD) Preparation and Maintenance Guide

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DEFINITIONS

Advanced Change Notice (ACN) – Documentation of major change(s) to a PCD prior to a general PCD revision. NCAMP approval is required.

Carbon Fiber Lot – see applicable NMS specifications.

Controlled Process Equipment (CPE) – Critical manufacturing equipment approved by NCAMP for the production of carbon fiber. Changes to the equipment may affect the quality of the carbon fiber.

Controlled Process Parameters (CPP) – Critical manufacturing process parameters approved by NCAMP for the production of carbon fiber. Changes to the parameters may affect the quality of the carbon fiber. CPP is also known as key process parameters (KPP).

General PCD Revision – A general revision to the PCD to incorporate all past major and minor changes.

Process Control Document (PCD) – An NCAMP-approved material manufacturer proprietary document which describes the "recipe" for the production of NCAMP carbon fiber material specification. It includes details of such items as raw material names or codes, raw material sources, raw material control, significant items of processing equipment, process flow, controlled process parameters, process tolerances, and process test information.

Supplier Review Board (SRB) – A committee established by the material supplier/manufacturer whose purpose is to review and disposition materials which have minor discrepancies.

1. Introduction

A carbon fiber process control document (PCD) is a "recipe" for the production of carbon fiber that conforms to NCAMP Material Specification (NMS) requirements. It is used in conjunction with carbon fiber material specifications for the purpose of controlling the carbon fiber material properties and to manage changes.

This document serves as a guide for carbon fiber manufacturers to prepare a polyacrylonitrile (PAN) -based carbon fiber PCD. This guide has been reviewed by all NCAMP members, including the FAA, DoD, aircraft companies, prepreg companies, and carbon fiber manufacturers. Unlike the NCAMP prepreg PCD review process, NCAMP will not be responsible for arranging carbon fiber PCD review for material users and prepreggers. Material users and prepreggers who wish to review the PCD should make arrangement directly with the carbon fiber manufacturer. NCAMP staff will review the PCD and make sure that the PCD is written in accordance with this guide prior to signing the PCD on behalf of the material users. This guide serves as a "bridge" between material users (or prepreggers) and carbon fiber manufacturers for the purpose of reviewing and approving carbon fiber PCD. NCAMP will be responsible for coordinating the review process of carbon fiber material specifications with all participants including material users and prepreggers.

There are many rules associated with the creation, approval, usage, and maintenance of a PCD. Because a carbon fiber PCD by nature is a proprietary document and the intention of the PCD review process is to ensure adequate material and process control, sensitive information may be coded or normalized. Whenever possible, requirements that do not involve proprietary information should also be listed in applicable material specifications. Only information that is proprietary in nature should be listed in the PCD only, because a PCD is less accessible to the material users.

2. Carbon fiber PCD Review and Approval Process

The carbon fiber PCD review and approval processes will involve the carbon fiber manufacturer and NCAMP staff. The carbon fiber manufacturer is required to prepare a draft carbon fiber PCD for each fiber and ensure that:

a) The PCD has adequately specified all requirements necessary to ensure proper control of the carbon fiber material properties.
b) The content of the PCD follows the requirements of section 4 below and the corresponding NMS material specification requirements.

The manufacturer and NCAMP staff must make sure that the PCD requirements are in agreement with the corresponding NMS material specification requirements.

3. Facility Audit

A facility audit may be conducted by NCAMP staff. Facility audits typically include:

a) Review of manufacturing facility, where the manufacturer must be able to show that the PCD(s) and applicable material specification(s) are followed, and
b) Review of manufacturer quality system.

4. Content of a PCD

A PCD should describe the raw ingredients, consumable materials, and the entire manufacturing process. Specifically, the PCD should include the following information:

4.1 Raw Ingredients and Consumable Materials

a. Specify all raw ingredients and consumable materials including procurement specifications/documents, approved manufacturers, and their addresses. Due to the proprietary nature of the PCD, the names of the ingredients may be coded.
b. Provide PAN lot definition. Define PAN fiber shelf life, packaging/storage, and marking requirements. List the quality control test requirements on the PAN fiber. Although a PAN precursor PCD is not required, changes to the PAN precursor, as described in section 6 below, will require NCAMP approval.
c. Certification tests, handling, storage requirements, mixing, and in–process test(s) for the sizing shall be documented.
d. For other raw ingredients and consumable materials, specify packaging, storage condition, shelf-life, and marking requirements. Specify shelf-life revalidation procedures, if applicable.

4.2 Manufacturing Process and Control

a. Employee training and qualification requirements shall be described in the PCD or related document. It is advisable that employees be briefed through a meeting by an engineer or manager in charge prior to each manufacturing process run to ensure that they are fully aware of the preparation procedures, process parameters, set-points, and the requirements of the PCD. Procedures to check equipment calibration dates and raw ingredients and consumable materials expiry dates should be part of every preparation process.
b. Describe the general manufacturing process and procedures encompassing oxidation, carbonization, graphitization, surface treatment, sizing application, and collection processes. Specifically:
    i. Provide diagrams to show the equipment and settings, the flow of the manufacturing process, including where in-process monitoring, inspection, and testing takes place.
    ii. Identify all Controlled Process Equipment (CPE) by line, model, and serial numbers.
    iii. Define all Controlled Process Parameters (CPP) along with their target values and tolerances. For example, provide the target value and tolerance on temperature for each manufacturing step. The values and units (e.g. °F) may be coded or normalized to a fictitious unit system (e.g. percentage) to protect proprietary information. In general, the tolerance must be approximately plus or minus six times (no more than seven times) the standard deviation of a normally

operating equipment/process (i.e. variation due to natural, non-assignable causes), unless a wider tolerance may be substantiated. If the process parameters fall outside the tolerance limits, the carbon fiber lot must be sub-lotted into inspection lots within which the parameters must be within the acceptable ranges. Non-conforming material must be handled per section 5 below.

iv. Where applicable, statistical process control techniques must be utilized to detect out-of-control process using control limits. Criteria to take action must be established, for example, a single point outside control limits, two consecutive points near control limits, five consecutive points above or below target value, etc. Action to look for and correct assignable causes should be taken when out-of-control process is detected. In addition, the production set-points must always be the same as the target values; production must not intentionally use other target values even though they are within the allowable tolerance limits, unless for reasons listed in vi. below.

v. Specify the sampling plan. Samples must be taken to be representative of the width of the production line or taken randomly from various creel or winder positions. In addition, the samples must be taken to be representative of the entire time of the production lot run.

vi. The carbon fiber properties are the result of cumulative time, temperature, and chemical/gas exposure history from all the process steps. As a result, not all combinations of upper or lower tolerance limits will yield acceptable carbon fiber properties. The manufacturer should be familiar with the cumulative effects of every process on the carbon fiber properties in order to fine-tune process set-points. The fine-tuned process set-points must be within PCD limits.

vii. Transition materials produced in non-steady-state conditions, such as those during start-ups or interruptions, shall be discarded. Define the criteria for determining and procedures for discarding transition materials.

viii. Specify tow splicing process and equipment.

ix. Define the carbon fiber tow down-spooling requirements. Down-spooling procedures, equipment, additional tests, and inspection frequency shall be documented in the PCD. Describe acceptance inspection procedures used to evaluate the finished carbon fiber and state the acceptance/rejection criteria. In addition, down spooling operation shall not change twist characteristics or sizing level.

x. Define the carbon fiber tow re-spooling requirements. Re-spooling procedures, equipment, additional tests, and inspection frequency shall be documented in the PCD. Describe acceptance inspection procedures used to evaluate the finished carbon fiber and state the acceptance/rejection criteria. In addition, re-spooling operation shall not change twist characteristics or sizing level.

xi. Define surface treatment method and level. Define sizing type, sizing level, and drying methods.

xii. Describe any foreseeable rework procedures, if applicable.

c. Where applicable, tolerance should be set based on Type I error probability of 1 percent with one retest allowed as described in section 6 of DOT/FAA/AR-03/19. Where such

method of establishing tolerance is inappropriate, the tolerance must be reasonable and able to provide adequate controls.

d. Where applicable, alarm devices must be present to ensure that operators are alerted when actual process measurements fall outside the permissible tolerance limits.
e. Actual in-process measurements must be taken at proper intervals and become part of the manufacturing records.
f. Manufacturer shall maintain records of all carbon fiber lots traceable to the PAN lot and doff numbers along with all relevant certification, Statistical Process Control (SPC), inspection, and manufacturing records for a minimum of seven years.

4.3 Approval of Raw Ingredient Manufacturers During Initial Qualification Program

This section describes the guidelines for the approval of raw ingredient manufacturers during the initial material qualification process. In general, the raw ingredient manufacturers used to fabricate the original qualification fiber are considered approved manufacturers (original qualification fiber are those used in the production of original qualification prepreg). Up to three manufacturers of the same raw ingredient may be included in the PCD if:

a. The raw ingredient (excluding PAN fiber) from the second and third manufacturer is chemically and physically *identical*, which may be difficult to prove. There shall be compelling data verifying the alternate material is identical to the original material, *or*
b. Prior evaluation suggests that the raw ingredient from the second and third manufacturer is chemically and physically *similar* and each source of the ingredient is used to produce at least one fiber lot of qualification material.

The use of more than one raw ingredient manufacturer may increase batch-to-batch variability if the materials are not truly identical. If significant batch-to-batch variability is detected during the qualification program, the extent of which the second and/or third raw ingredient had influenced the variability must be investigated.

4.4 Approval of Controlled Process Equipment During Initial Qualification Program

This section describes the guidelines for approval of Controlled Process Equipment during the initial material qualification process. In general, the equipment used to fabricate the original qualification fiber are considered approved equipment. If second or third equipment is desired, it may be included in the PCD if:
a. The second or third equipment is *identical* and is not primary fiber production line equipment. Evidence to prove that the second or third equipment is identical is required, or
b. Prior evaluation suggests that the second or third equipment is similar and output is identical, and the equipment is used to produce at least one fiber lot of qualification material. NCAMP must be involved in the evaluation process.

The additional equipment cannot be allowed if:
a. The equipment changes the time, speed, temperature, and chemical/gas exposure history of the fiber.
b. The equipment is expected to change any characteristic of the material.

Using more than one piece of equipment may increase batch-to-batch variability if the equipment is not truly identical. If significant batch-to-batch variability is detected during the qualification program, the extent of which the second and/or third equipment had influenced the variability must be investigated.

5. Non-Conforming Material and Disposition

5.1 Minor Discrepancy

Carbon fibers which do not conform with the PCD may be regarded as having minor discrepancies provided that the carbon fibers are produced using approved Controlled Process Equipment and meet all applicable Controlled Process Parameters and NMS requirements. It is recommended that material manufacturer establish a Supplier Review Board (SRB) to evaluate the minor discrepancy. SRB is a committee established by the material manufacturer which consists of at least two senior members with the purpose of reviewing and dispositioning materials which have minor discrepancies. At least one member of the SRB must have an in-depth understanding of the product chemistry and possible impact on the product usability and material property. "Use-as-is" disposition must be justified by test and/or past experience (one retest is typically allowed). Every member of the SRB must agree that the discrepancy has no effect on the carbon fiber's properties and handling qualities, and the disposition must be signed by all the SRB members. The material shall be regarded as having major discrepancy if at least one SRB member disagrees. Manufacturer that does not have a SRB will not be allowed to disposition minor discrepancies; all discrepancies must be considered as major discrepancies.

5.2 Major Discrepancy

Carbon fibers that fail to meet NMS requirements, produced outside of Controlled Process Parameters, or produced using unapproved Controlled Process Equipment are regarded as having major discrepancies and shall be segregated. There must be justification (typically backed by evidence such as in-process monitoring data or test data) to ensure that the discrepancy does not extend into accepted carbon fibers.

The carbon fibers may be reworked (if applicable) and accepted in accordance with the procedures in PCD or written instructions of the SRB, provided that:

a. Traceability is maintained for all rework procedures, if rework is performed.
b. Compliance with applicable NMS requirements is demonstrated through subsequent acceptance testing and/or inspection.
c. Every member of the SRB agrees that the rework procedures (if applicable) have restored the carbon fiber properties and subsequent testing and/or inspection have yielded acceptable results.

Otherwise, the carbon fiber shall not be certified to the NMS material specification.

6. Revisions and Change Control

The manufacturer shall maintain a good record of all PCD revisions and associated ACN and minor changes and be able to reconstruct the PCD to any point in the last seven years.

Minor Changes – Changes that do not affect the materials or processes. Minor changes can be divided into Level 0 and 1 changes as described below.

Level 0 Changes – These are changes such as typographical error corrections, changes to the names of incoming materials due to company name changes, and use of alternate storage facility locations using identical storage conditions. No notification to NCAMP or the purchaser is required.

Level 1 Changes – These changes typically involve the use of an *identical* auxiliary material or equipment. An example is an alternate vendor for chemically and physically *identical* auxiliary raw material (there should be compelling data verifying that the alternate material is identical to the original material). Another example is the repair or replacement of a part within the fiber manufacturing line. Minor revisions to the PCD or related documents that involve reducing the tolerances to improve quality may also be regarded as Level 1 changes. These changes must be evaluated and/or tested internally beyond normal batch acceptance testing and have been found not to affect the material. Every member of the SRB (if SRB is available) must agree that there is no possible impact on the product usability and material property; otherwise, it should be treated as a Level 2 change. Notification to purchaser or NCAMP for Level 1 change is optional and at the discretion of the manufacturer.

Major Changes – Changes in the content of PCD which might affect the quality of the carbon fiber. NCAMP approval is required, and is granted through an ACN per section 6.1. Changes that may be regarded as major include, but are not limited to, the following:

- PAN Precursor formulation (raw ingredients and ratios)**,
- PAN Precursor manufacturing process, equipment, line, or site**,
- Widening of PAN Precursor acceptance requirements**,
- Carbon fiber tow processing parameters (e.g. temperature and speed),
- Carbon fiber tow manufacturing equipment, line, or site,
- Widening of carbon fiber tow acceptance requirements,
- Carbon fiber tow acceptance test methods,
- Carbon fiber tow acceptance sampling plan,
- Carbon fiber tow surface treatment methods and levels,
- Carbon fiber tow sizing formulation and sizing level, and
- Carbon fiber tow sizing application and drying methods including equipment.

** Although PAN precursor PCD is not required, PAN precursor is under change control as specified in this document.

Major changes can be further divided into Level 2, 3, and 4 changes. The following paragraphs describe the testing and notification requirements associated with these levels.

Level 2 Changes - These changes typically involve the use of *similar* material, equipment, or process. At minimum, tests must be conducted to an extent that establishes the requirements listed in the carbon fiber material specifications will not change. Additional testing at the carbon fiber, prepreg, lamina, or laminate levels may also be required depending on the extent of the change. Examples of level 2 changes include (1) Second source of chemically and physically *similar* auxiliary raw materials, (2) Addition of *similar* auxiliary equipment, (3) Modifications to process equipment or processes that do not change material key characteristics (KC, see material specification) or CPP, and (4) Expansion of existing facilities, including start-up of additional auxiliary production machines. It is recommended that side-by-side testing of the original material or method, and the new material or method, be conducted.

Level 3 Changes - These are changes that are subjected to an equivalency test program, such as the one defined in DOT/FAA/AR-03/19. Level 3 major changes are those that have the possibility of changing either the part processing characteristics or the cured lamina/laminate properties such that there is a shift away from the average values established for the material. Typical examples of level 3 changes are (1) Change in fiber manufacturing process such as speed, (2) Change in fiber size type, size level, or finish, (3) Change in manufacturing site for fiber, and (4) Change in nominal number of fibers per tow (small, difference of less than 200 fibers per tow).

Level 4 Changes – These are changes where equivalency tests will not suffice for links to a previous material characterization. Level 4 changes require a new product identification (i.e. a new specification designation), and a new qualification test program. NCAMP will not approve Level 4 changes through an ACN. Level 3 or lower material changes that fail to demonstrate equivalency will typically be considered as Level 4 changes. Typical examples of Level 4 changes are (1) Change in nominal number of fibers per tow (e.g., 3K fibers per tow to 6K), (2) Change in fiber type (e.g., T300 to AS4), (3) Change in fiber manufacturer (e.g., Toray to Cytec). Because level 4 changes are considered a new material, existing end-users will not be affected unless they elect to purchase the new material. An end-user who wishes to use the new material must perform sufficient tests to qualify and certify the use of the material in the intended application; an activity outside the scope of NCAMP.

6.1 Advanced Change Notice (ACN)

ACNs are interim documents used to approve major changes to the PCD. ACNs will be approved only after the major changes have been shown to have negligible effect on the carbon fiber tow, prepreg, and composite material properties. Appendix 1 of this document contains the ACN form. Prior to implementing a major change, the manufacturer shall notify NCAMP and/or the direct customers (typically, prepreg companies or fabric weavers) with a detailed description of the proposed change(s) along with test results (if available) which

may substantiate the change. Since the detailed description may be sent to aircraft companies that may not have direct non-disclosure agreement (NDA) with the carbon fiber manufacturer (although the aircraft companies typically have NDA with the prepregger), certain proprietary information may be coded or omitted, but the remaining information must be able to provide a thorough understanding of the change.

Since the original material qualification and allowables are performed at composite prepreg, lamina, and laminate levels, a majority of the material equivalency test matrix designed to substantiate the changes may also be conducted at these levels. The change will be reviewed and approved in accordance with NCAMP Recommended Practice (NRP) 101 "Prepreg Process Control Document (PCD) Preparation and Maintenance Guide." NCAMP may request the fiber manufacturer to perform tests at the carbon fiber level and provide samples of the new and/or old carbon fiber. Once the change is approved per NRP 101, NCAMP will sign the ACN. The signed ACN is the formal approval of the change and becomes part of the new PCD. The major change shall not be implemented until the ACN is signed.

6.2 General PCD Revision

Manufacturer will revise the PCD to incorporate all past ACNs and minor changes when there are a significant number of changes which make the document difficult to follow. A new PCD revision is also warranted when there is a revision to the corresponding material specification. The content of all Major and Minor Changes made to the PCD during the general PCD revision shall be summarized in the revision control block. Whenever possible, major changes should be proposed and approved through ACNs because the process requires involvement of other NCAMP members.

7. Other Requirements

The corresponding material specification will contain additional requirements. Manufacturers shall determine that the PCD is tailored to meet the material specification requirements.

8. References

1. Boeing D6-53356 Requirements for Process Control Documents for Suppliers of Nonmetallic Raw Materials, Rev NEW

2. FAA/DOT/AR-02/109 – Guidelines and Recommended Criteria for the Development of a Material Specification for Carbon Fiber/Epoxy Unidirectional Carbon fibers

3. NCAMP NRP 101 Prepreg Process Control Document (PCD) Preparation and Maintenance Guide

9. Revisions

Revision	Date	Description
-	March 27, 2017	Initial Release

APPENDIX 1: ADVANCED CHANGE NOTICE (ACN)
National Center for Advanced Materials Performance (NCAMP)
*** CONFIDENTIAL WHEN COMPLETED ***

To be completed by material manufacturer:

ACN NO.: DATE:	REQUESTED BY:
MATERIAL SPECIFICATION NO.:	SECTION TO BE CHANGED:
REASON FOR CHANGE:	
CURRENT DOCUMENTATION:	
PROPOSED CHANGE:	
COMPANY:	BY: _____ (TECHNICAL) _____ (OPERATIONS/PRODUCTION) _____ (QUALITY ASSURANCE)
COMMENTS:	

To be completed by NCAMP:

NCAMP STAFF:	LEVEL OF CHANGE:
APPROVED ( ) BY: _____ DATE: _____ *(upon reaching consensus with material users only)* DISAPPROVED ( ) TITLE: _____	
DATE OF INCORPORATION:	
COMMENTS:	