Statement of Work for X-Ray Diffraction System

I. INTRODUCTION

The contractor shall provide all personnel, equipment, and supplies necessary to fabricate, install, demonstrate, and provide operational training for a complete x-ray diffraction system. The capabilities of this system shall include, but not be limited to, the following: identifying phases and precise lattice parameter measurements in metals, intermetallics, and semi-crystalline polymers by powder diffraction, and identifying texture in metals and intermetallics.

Two optional sample stages and associated software are also requested to be priced separately: (1) adding the capability to measure residual stresses, and (2) adding the capability to conduct x-ray diffraction tests at elevated temperatures in an inert, air or vacuum environment. Any hardware and software required for the two options shall be fully compatible for integration with the base x-ray diffraction system.

Goniometer Geometry Conventions Used in the Statement of Work

The following conventions are used throughout this Statement of Work to identify the geometries associated with x-ray diffraction systems. The ψ-goniometer convention (American convention) uses ψ and χ designations for tilt and φ for rotational angles, and is the convention used in this Statement of Work. The ω-goniometer convention (European convention) uses ω and ψ designations for tilt and φ for rotational angles. The ω-goniometer designations for tilt angles are listed in parentheses immediately following the ψ-goniometer designations. In both ψ- and ω-goniometer conventions, φ designates a rotation in the plane of the specimen surface. While X and Y designate two perpendicular directions in the plane of the specimen, Z designates the normal to the specimen plane.

II. X-RAY DIFFRACTION SYSTEM REQUIREMENTS (CLIN 1)

1.0 GENERAL REQUIREMENTS

1.1 The contractor shall deliver an x-ray diffraction system that can be reconfigured by the users to perform powder diffraction and texture tests. The system shall be modular in design to allow for future expansion. All structure, plumbing, wiring, cabling, valving, cooling, controls, and safety devices for safe, research-oriented operation shall be included.

1.2 Powder Diffraction – The x-ray diffraction system shall perform standard powder x-ray diffraction scans on metallic, intermetallic and semicrystalline or multiphase polymeric materials, and precision lattice parameter determination in metallic materials. Metallic materials include aluminum, titanium, nickel, and ferrous-based alloys. X-ray diffraction scans over a small (0.5^o to 40^o) 2θ range shall be required for polymeric materials, and scans over a larger (5^o to at least 165^o) 2θ range shall be required for metallic materials. This system shall also include search-match and database software required for identifying phases in metals, ceramics, intermetallics, and semi-crystalline polymers.

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1.3 Texture – The x-ray diffraction system shall perform pole figure determination and orientation distribution functions (ODF) texture analysis on metallic materials such as aluminum, titanium, nickel and ferrous-based alloys, and intermetallics. These materials may have large grain sizes and/or high degrees of preferred orientation, and the sample sizes and shapes may vary considerably. These tests will require high angular precision, accuracy, and a large number of measurements.

2.0 SPECIFIC REQUIREMENTS –

2.1 The x-ray diffraction system shall include:

2.1.1 Power – The x-ray diffraction system shall be capable of full, safe, and controlled operation using the following utilities available in the designated laboratory location at Langley Research Center: 60 ampere / 208 volt / 60 Hz / 1 phase AC; and 15 ampere / 125 volt / 60 Hz / 1 phase AC.

2.1.2 Water Cooling – All closed-loop water chiller/heat exchanger and necessary plumbing, valving, and monitoring devices required for full, safe, and controlled operation of the x-ray diffraction system shall be included.

2.1.3 X-Ray Tubes – The x-ray diffraction system shall include, as a minimum, one copper and one chromium x-ray tube, each of which is capable of performing powder diffraction on materials including semi-crystalline polymers, aluminum, titanium, nickel, and ferrous-based alloys and intermetallics. In addition, the focus of the tubes shall be capable of performing high resolution powder diffraction, texture, and residual stress measurements. The x-ray tube controls shall include an automatic mode to provide a controlled bake-out and conditioning of the tube for increased tube life.

The x-ray tubes shall be suitable for installation and removal by NASA personnel in our laboratory at Langley Research Center without requiring special tools. The x-ray tubes shall be interchangeable, and shall include all necessary mounting flanges, fixtures, and cooling plumbing required for full, safe, and controlled operation.

2.1.4 Beam Collimation – The system shall include a range of slits to control sample illumination, axial divergence, and resolution. The slits shall be capable of both manual, and computer adjustment and control to attain the resolution described in Section 2.1.5. A phosphorescent screen or other device shall be included to allow visual imaging of the beam for diagnostics purposes.

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2.1.5 Resolution - The resolution of the x-ray diffraction system shall have a minimum step size of 0.0001° 2θ or smaller with a repeatable peak position accuracy of at least ±0.01° 2θ over the total angular range. The total 2θ angular range over which data can be collected shall be 0.5° to at least 165°. The precision of the x-ray diffraction system shall produce symmetric peaks with the following peak moment statistics: variance < 0.001 degrees squared; skewness < 0.2; and excess < 1.

2.1.6 Alignment and Calibration – The x-ray diffraction system shall be designed so NASA personnel can perform precise alignment and calibration within the resolution described in Section 2.1.5. Positioning of the samples shall be within 0.025 mm in each linear direction, and within 0.01° in each angular direction.

2.1.7 Primary Detection System – The x-ray diffraction system shall include a means for rapid detection of x-rays (attaining resolutions in accordance with Section 2.1.5 with a total scan time of less than 4 hours), which is suitable for performing high resolution powder diffraction tests in accordance with Section 1.2 of this Statement of Work, and texture tests in accordance with Section 1.3 of this Statement of Work. The detector shall be interchangeable with the back-up detector (see Section 2.1.8), and shall require no specialized tools for changing other than those supplied with the system. The hardware, electronics, power supplies, and monitoring devices required for full, safe, and controlled operation shall also be included.

2.1.8 Specimen Size – The x-ray diffraction system shall be capable of measuring textures on specimens with dimensions of at least 2.5 cm x 2.5 cm x 0.5 cm and weight of at least 0.5 kg, without compromising tilting, rotational, and sample positioning capabilities. Powder diffraction capabilities shall be capable of handling powdered samples and small solid samples with dimensions of 4 mm x 4 mm x 0.5 mm or smaller. The pole figure shall be a bulk measurement acquired over a 1.5 cm x 1.5 cm area located in the center of the 2.5 cm x 2.5 cm sample.

2.1.9 Sample Positioning – The x-ray diffraction system shall have the capability of specimen translation to map residual stresses and textures across the surface of a specimen. All hardware, mounting fixtures, and software required to achieve sample positioning shall be included, and shall be compatible with residual stress and texture operation. There is the requirement for continuous spinning of the sample in addition to 360° φ-rotation described in 2.1.10.

2.1.10 Sample Holding and Spinning - The x-ray diffraction system shall include two different sample holding methods:

a. x-translation of the sample, and
b. 360° φ-rotation in the specimen plane, for all applications

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These methods shall be compatible with standard sample averaging techniques to increase the areal exposure of the sample through translation or spinning, as well as stationary operation. A zero-background holder shall be included for measurement of small samples measuring 4 mm x 4 mm x 0.5 mm or equivalent quantities of powdered samples.

2.2 Back-Up Detector System - A proportional or scintillation detector shall be included as a back-up detector. This detector shall be mountable onto the primary detector couplings and shall include all associated hardware, electronics, power supplies, and monitoring devices required for full, safe, and controlled operation.

2.3 Computer Controller/Data Acquisition System - The x-ray diffraction system shall be controlled by a Windows based computer with an operating system of Windows 7 or higher. The computer shall control and monitor the x-ray diffraction system and shall allow safe, unattended operation (see Section 2.5). The computer shall be fully loaded with all required software to control the x-ray diffraction system, collect and store all data, and perform all analyses as described in this Statement of Work. The computer shall include a CPU, keyboard, mouse and monitor. The CPU shall be equipped for the following performance, as a minimum: Core 2 dual i5 processor (2.8 GHz), 8 Gb RAM, 500 Gb hard drive, 48x DVD R/W. This computer and all peripherals shall be supplied by the contractor.

2.4 Software - The x-ray diffraction system shall include software for automated system control of the x-ray diffraction tests, compatible with the Windows 7 operating system or higher. Data shall be accessible as Microsoft Excel-compatible data files and transportable to another computer for further data reduction. Software shall also include licenses to permit installation of the data reduction software on up to five (5) additional computers not including the system control computer, to enable data analysis to be performed in other locations. All of these computers will reside at Langley Research Center. Due to computer firewall issues, the software must be operable in a stand-alone mode, without requiring access to external computers or web-based programs.

2.4.1 Powder diffraction data reduction software shall include, but not be limited to, calculation of d-spacing, phase identification, and peak deconvolution. Software shall include a complete diffraction library for inorganic materials, and routines for performing search-match against the library.

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2.4.2 Texture software shall be capable of generating standard pole figures. Pole figure contour lines shall be marked with times random units and relative intensities. Texture software shall also be capable of manipulating raw data to produce polar plots, pole figures, and automatic conversion of that data into conventional ODF plots, with output available in either the Bunge or Roe convention or both.

2.5 Safety – All OSHA radiation health and safety specifications shall be met. Safety features shall allow safe, unattended operation with interlocks that will activate automatic shutdown in the events of: power failure, loss of coolant flow, removal of safety shielding during operation, opening of system access doors, or any other potentially hazardous situations that could cause exposure to harmful radiation levels. The safety shielding shall have doors that allow full access to the interior for system adjusting and sample changing when x-rays are not energized.

3.0 INSTALLATION AND VERIFICATION
The contractor shall provide the necessary field engineers/technicians to install, inspect, and verify that all components of the x-ray diffraction systems are in good working condition and operating properly at the designated site at Langley Research Center. In addition, the contractor shall successfully perform acceptance testing, as described in Section 4.0, on-site at Langley Research Center.

4.0 ACCEPTANCE TESTS
Samples, as indicated below, used in the Final Acceptance Tests, will be provided by the Government at the initiation of acceptance testing.

4.1 Resolution and Peak Symmetry - The contractor shall demonstrate the resolution capabilities of the system on a LaB_6 specimen (NIST SRM #660) or fine grained Alumina (Al_2O_3) specimen (NIST SRM #1976). Statistics on the peak moments shall be used to assess the symmetry of these peaks. The peak moments shall have the following statistics.

Variance < 0.001 degrees squared
Skewness < 0.2
Excess < 1

4.2 Sample Positioning - The contractor shall demonstrate the ability to obtain x-ray diffraction data from any x-y point on a sample with dimensions of 2.5 cm x 2.5 cm x 0.5 cm. The sample will be a section from standard commercially-available 316 stainless steel rolled sheet and can be provided in advance. The hardware configuration to be tested shall be whatever configuration is proposed for conducting powder diffraction experiments described in section 1.2 of the Statement of Work. Acceptance criteria is <2% 2θ variation on all peaks for measurements taken at each corner and in the center of the sample provided.

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4.3 Sample Averaging – For all sample holders described in Section 2.1.10: On a large-grained metal specimen (such as annealed commercially pure titanium), the contractor shall demonstrate that the sample averaging devices are effective in enhancing the diffraction spectra by performing scans with and without the sample averaging device active and comparing the resulting Intensity vs. 2θ data. The sample will be a 2.5 cm x 2.5 cm section from a large-grained piece of annealed titanium (the grain structure is readily visible with the unaided eye, with a columnar grain structure with grains 2-4 mm in width and length that runs the complete length of the 2.5 cm dimension on the specimen). The sample can be provided in advance. The hardware configuration to be tested shall be the configuration being utilized for conducting powder diffraction experiments described in section 1.2 of the Statement of Work. The test will involve taking one powder diffraction scan (10-140° 2θ) measurement with a finely collimated x-ray beam and the sample stationary, and a second powder diffraction scan over the same range and with the same collimation but with the sample average device activated. Acceptance criteria is demonstrating all peaks match the anticipate 2θ location and relative intensity (as identified in the International Centre for Diffraction Data (ICDD) standard database for titanium) in the 2θ scan taken with the sample averaging device activated. This data shall show an improvement over the 2θ scan taken while the sample is held stationary (i.e. in the stationary sample, peaks will be missing and relative intensities of different peaks may be incorrect due to the large grain structure and texture in the sample). These methods may include, but not be limited to, continuous sample spinning or oscillating the sample in the X or Y direction (in the plane of the sample surface).

4.4 Texture - Pole figure determination and ODF analysis shall be performed on a rolled aluminum sheet specimen with documented texture to demonstrate the texture techniques, hardware and data reduction capabilities and accuracy of the measurement and data reduction system. The sample shall be a 2.5 cm x 2.5 cm section from a rolled Al-Li sheet with texture that has been verified by other means (such as microscopically with Electron Beam Backscattered Detection in a scanning electron microscope). The sample can be provided in advance. The hardware configuration to be tested shall be the configuration proposed for conducting texture experiments described in section 1.3 of the Statement of Work. Acceptance criteria is generation of pole figures and ODF analysis that are consistent (<10% variance) with the texture measured on the same sample by other means.

5.0 MAINTENANCE/WARRANTY

The contractor shall provide an extended maintenance/warranty of five (5) years after acceptance of the system. This shall cover all parts and labor, excluding standard consumables (such as lubricants, disposable sample stages, and x-ray tubes). This shall also include a yearly preventative maintenance (PM) service performed on-site at

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NASA Langley, including annual checks of alignment, cleaning, lubrication, installation of software upgrades, and overall system check. The warranty shall also include all available software upgrades for the five (5) year period.

6.0 DELIVERABLES

1) X-Ray Diffraction System including all software and the backup detector system, in accordance with the specifications in this Statement of Work.

2) One (1) Windows based computer for complete control, operation and data analysis with the X-Ray Diffraction System in accordance with the specifications in this Statement of Work.

3) Operation/Maintenance Manuals - Two complete sets of operating manuals shall be provided (either hard copy or on a DVD CD-ROM). These manuals shall contain detailed operation, maintenance, and trouble-shooting instructions for all system configurations and components. These manuals shall also include electrical wiring diagrams of the x-ray diffraction systems and components, as well as schematic diagrams of the components showing the pertinent physical characteristics, including any on-site hardware modifications.

4) Software Manuals - Two complete sets of software manuals shall be included (either hard copy or on a DVD or CD-ROM). These manuals shall contain documentation indicating conventions, corrections, and associated equations used in the software for the data reduction routines, explicit descriptions of data-file architecture, and non-proprietary portions of the source code. These manuals shall also contain detailed operation, maintenance, and trouble-shooting instructions for the software operation of the x-ray diffraction systems and attachments, including any on-site software modifications. In addition, one copy of the software is required on a DVD or CD-ROM.

7.0 SCHEDULE

Delivery of all equipment, software and manuals shall be made to NASA Langley Research Center with 150 days after the contract award date. Installation of all equipment shall be within 30 days after delivery of all equipment and software.

III. TRAINING (CLIN 2)

The contractor shall provide the necessary personnel to train, at Langley Research Center in Hampton, VA, up to ten (10) Langley personnel in the operation, trouble-shooting, and maintenance of the systems. The training period shall continue for five (5) full working days after satisfactory completion of the final acceptance tests (See Section 4.0). In addition, one week of specialized training shall be included for two (2) operators, to be completed within one (1) year following the on-site training as described above. The specialized training may be conducted at the contractor's

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facility and shall consist of more in-depth data collection, analysis and specialized techniques including but not limited to texture, powder, residual stress, and elevated temperature tests.

IV. OPTION 1: (CLIN 3)

1.0 RESIDUAL STRESS

This option shall include any additional hardware or software necessary to expand the capability of the base x-ray diffraction system to enable residual stress measurements.

This option shall allow the Government to routinely perform residual stress measurements on metallic materials such as aluminum, titanium, nickel, and ferrous-based alloys and intermetallics. These materials may have large grain sizes and/or high degrees of preferred orientation, and the sample sizes and shapes may vary considerably.

X-ray diffraction scans shall be possible over a range of 126^o to 165^o 2θ. The system shall be capable of measuring residual stresses on specimens with dimensions of at least 20 cm x 10 cm x 5 cm and weight of at least 1 kg, without compromising tilting, rotational, and sample positioning capabilities.

This option shall also include all necessary software to set up, perform, and evaluate residual stress measurements using the $\sin^2\psi$ convention. Residual stress data reduction software shall be capable of calculating uniaxial, biaxial, triaxial, and shear stresses and the geometric and counting statistics errors associated with each of these stress states.

1.1 Acceptance Test: Residual Stress - The uniaxial, biaxial, triaxial, and shear residual stress states shall be determined in an aluminum specimen with documented residual stress to demonstrate the residual stress techniques, hardware, and data reduction capabilities and accuracy of the measurement and data reduction system. The sample will be a 15 cm x 5 cm x 2.5 cm 2219 aluminum sample with a welded bead down the center that has documented residual stresses (measured by neutron diffraction). The sample can be provided in advance. The hardware configuration to be tested shall be the configuration proposed for conducting residual stress measurements described in section IV. Acceptance criteria is measuring residual stresses in the specified locations on the sample that are consistent (<10% variance) with the surface-measured residual stresses obtained by neutron diffraction. It is recognized that neutron diffraction provides deeper penetration than x-rays, so the stress values will be compensated to be representative of the depth of penetration of typical x-ray residual stress measurements.

V. OPTION 2: (CLIN 4)

1.0 ELEVATED TEMPERATURE X-RAY DIFFRACTION

The contractor shall supply any additional hardware and software necessary to

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expand the capability of the base x-ray diffraction system to enable x-ray diffraction scans at elevated temperatures in air, inert gas, or vacuum on polymeric and metallic materials.

Specifically, crystalline transitions in low-scattering polymeric films will be monitored. Since crystallization kinetics in these materials involve high transformation rates, rapid x-ray diffraction scans over a small (0.5^o to 40^o) 2θ range will be required to monitor crystallization during a heating profile.

A heating profile will include several heating rates, isothermal holds, and cooling cycles, and may be several minutes to several hours in duration. Also, crystalline transitions may cause shifts of only 0.05^o 2θ or smaller; or reduction of one set of peaks transitioning to a completely different set of peaks shifted by 10^o 2θ or greater in metallic material phase transformations.

1.1 Sample Positioning Geometry – The elevated temperature stage shall keep the specimen horizontal during 2θ scanning, to allow heating of specimens to their melting points without pouring the molten material off of the sample holder.

1.2 Elevated Temperature - The elevated temperature stage shall have the capability of heating a specimen over the temperature range of ambient temperature to 1200^oC. The heating rates shall be controllable from 5^oC/minute to 50^oC/minute or greater, while maintaining a ramp linearity of $\pm 2^oC$ or better over this entire range. The elevated temperature stage shall be capable of isothermal hold steps for 0 to 10 minutes or longer while maintaining $\pm 1^oC$. The elevated temperature stage and all associated hardware and electronics shall be modular in construction such that attaching to and detaching from the main x-ray diffraction system may be performed in our laboratory at Langley Research Center without requiring special tools.

1.3 Atmosphere - The elevated temperature stage shall have the capability of operating under a variety of atmospheric conditions, including vacuum (10^{-6} torr), air, and inert gases. The total pressure shall not exceed 760 torr. Hardware, electronics, controllers, and monitors required for obtaining and maintaining a vacuum of 10^{-6} torr or better shall be included and integrated with system computer controls. The controlled atmosphere attachment shall be modular in construction such that attaching to and detaching from the x-ray diffraction system may be performed in our laboratory at Langley Research Center without requiring special tools other than those supplied with the system.

1.4 Acceptance Tests

1.4.1 Elevated Temperature Accuracy - The contractor shall demonstrate isothermal temperature holds and temperature accuracy by showing the crystalline peaks of tin and/or gold 2^oC below their melting points, and then

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showing the absence of crystalline peaks 2°C above their melting points in inert gas or a vacuum environment.

1.4.2 Elevated Temperature Resolution - The contractor shall demonstrate the resolution of the x-ray diffraction system with the elevated temperature stage installed by measuring the thermal expansion changes in d-spacings on an aluminum sheet specimen in air. The contractor shall also perform x-ray diffraction scans on a lucite sample to demonstrate the system resolution and speed with an amorphous material.

1.4.3 Sensitivity and Peak Resolution - A series of x-ray diffraction scans (5° to 40° 2θ) shall be performed on a semi-crystalline polyethylene film during a 30-minute heating profile. Detection of incipient crystallization and the subsequent stages of phase transformations during the heating profile shall demonstrate the speed, sensitivity, and peak resolution capability of the detector in combination with the elevated temperature attachment.