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# RPPR Final Report
as of 09-Sep-2019

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Proposal Number: 70536RTREP **Agreement Number: W911NF-17-1-0451**

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**Title:** Acquisition of a Scanning Electron Microscope (SEM) for Multidisciplinary Research at Virginia State University
**Begin Performance Period:** 30-Sep-2017 **End Performance Period:** 29-Mar-2019
**Report Term:** 0-Other
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**STEM Degrees:** **STEM Participants:**

**Major Goals:** It is urgent to perform cutting edge research, educational training and outreach program in academic institutions under the category of minority-serving institution (MSI) and historically black colleges and universities (HBCU). Virginia State University (VSU) is a Historically Black University (HBCU) having both undergraduate and graduate engineering and science majors. Also, many interdisciplinary researches are on-going at the Virginia State University campus and with external collaborators.

The major goals of this proposal are:

1. Acquisition of Scanning Electron Microscope at VSU.
2. Establish a multi-user environment that provides technical support and user training and application expertise in support of individual user projects and enhances STEM educations at VSU.
3. To Introduce the concepts of science, technology, engineering, and mathematics (STEM) to college and high school students by giving them the opportunity to work with scientists/researchers on innovative concepts.
4. Exposure to state-of-the-art equipment and software to VSU students and local high school teachers and students.
5. Development of laboratory skills needed for success in future education thus making the high school students more competitive for college and enhancement of their critical thinking skills, and research interest in STEM disciplines.

SEM will be a common instrument and characterization tool of technical research and training that require imaging, and elemental analysis. Therefore, SEM will provide strong collaboration among faculties to enhance the outstanding research/teaching opportunities for STEM education. Furthermore, SEM is equally basic as well as a necessity to our Center for Undergraduate Research (CUR) at VSU for the continuing research and education program. Bringing together multi-disciplinary teams from the science, engineering and agriculture at VSU, this will significantly accelerate and foster integration of research & training and support our CUR. The SEM will also provide an opportunity for laboratory experience and training in image analysis projects for undergraduate and graduate students. The instrument will support a robust research program that provides unique experiences to faculties at VSU, and strong collaborations to our university and industry partners. The goal of this state-of –the-art instrument is to encourage faculty and students within the science, technology, engineering, and mathematics (STEM) community for interdisciplinary research, education, and outreach program.

Scanning Electron Microscope (SEM) performs a very important role in modern scientific research, especially in medicine, engineering, biology, chemistry, physics, material science and nanotechnology. VSU campus faculty research in polymer science, surface coating, nano/micro manufacturing, and image analysis are in line with our goal to align our activities with the Army Research Laboratory’s (ARL’s) Open Campus initiative program in future. Specifically, VSU is committed to this effort because of a close alignment with ARL’s campaign for Functional Integration of Materials and related capabilities to demonstrate predictive design and integration of functional properties into complex multi-component materials systems.
In addition to this another important goal is to use the instrument for research purposes and collect the scientific data which could be tremendous interest to DoD.

**Accomplishments:** Due to some technical circumstances, the installation of the SEM instrument has been delayed since several months. The engineers from JEOL started the assembly of the instrument from May 2019. Currently, the installation of the instrument is ongoing. However, the PI (Dr. Samantaray) and the faculty (Dr. Ghariban) were trained how to capture the high resolution image of the samples. We are planning to provide a complete training for other faculties during mid of July 2019.

Hydrogel Nanocomposites
Among all types of nanomaterials used for industrial and biomedical applications, magnetic iron oxide ($Fe_3O_4$), have been used in a wide array due to their unique properties at nanometer scale. Hydrogel nancomposite has been synthesized by incorporation of magnetic iron oxide nanoparticle by in-situ precipitation method in the hydrogel matrix.

Hydrogel Synthesis:
Hydrogels were synthesized by redox ploymerization method with varying the crosslinking densities. N-isopropylacrylamide (NIPAAm), Polyethylene glycol 200 diacrylate(PEG200DA) , Ammonium persulfate (APS), Tetramethylethylenediamine (TEMED), Iron (II) chloride tetrahydrate, Iron (III) chloride, Ammonium hydroxide, Phosphate Buffered Saline (PBS) were used for the nanocomposite hydrogel synthesis.
In a typical experiment, desired amounts of monomer and crosslinkers were mixed with ethanol. About 25wt% with respect to the total monomer and crosslinker amount ethanol solvent was used. To this solution 1wt% APS was added and the solution was mixed properly. Then, 10µL of TEMED was added to the monomer/cross-linker solution as well. The mixture was then pipetted into 15×15 cm2 clamped glass plates separated by 750 µm Teflon spacer. The polymerization was then carried out overnight. The gel was washed with deionized water several days in order to remove any unreacted products in the matrix. After that the gel was cut into desired size of discs and used for in situ precipitation of the nanoparticles in the matrix. Different grades of hydrogels (90:10, 80:20, 70:30 and 50:50) were synthesized by varying the molar ratios of monomer and crosslinker.
Nanocomposite Synthesis:

First the metal salt solutions are prepared by dissolving appropriate amount of FeCl3.6H2O and FeCl2. 4H2O in DI water so that the final molar concentrations of the salts are 2:1. The hydrogels with different crosslinking densities were soaked in this salt solution at 450C for 24 hrs. The hydrogels were washed thoroughly several times after that to remove any surface bound and unbound metal ions in the matrix. The gels were transferred to flask containing ammonium hydroxide (NH4OH) and the reduction process was continued for 24 hrs at 450C. Finally the gels were washed several times with DI water to remove the unbounded metal particles. The surfaces of the gels were cleaned with kimwipes in order to remove any excess unbound surface particles. The soaking in metal salt solutions and NH4OH was repeated 5 times to precipitate more iron oxide nanoparticles in the hydrogel matrix. The synthesized hydrogel s were characterized by FTIR, UV and XRD. The surface morphology and the nanoparticles formation were analysed by using our new partially installed SEM. With this new high resolution SEM, we were able to see the nanoparticle formation inside the hydrogel matrix. Figure 1-3, shows the SEM images of hydrogel nanocomposites (attached file in upload section). From the SEM images we confirmed that the nanoparticles were successfully synthesized inside a hydrogel matrix by in-situ method and the paticles are uniformelly distributed in the hydrogel matrix.

Microscopic Study for Additive Manufacturing

Additive manufacturing is a promising technology for the production of components for different industries. Its layer-by-layer build-up process offers a number of complex production possibilities. Among the different types of the additive technologies laser powder bed fusion (LPBF) has shown superiority in manufacturing parts with high level of dimensional accuracy, geometry complexity, surface quality; and inner structures. The powder profile characteristics such as particle shape, particle size, and particle size distribution are deriving factors of the material properties of the final product. A better understanding of the correlation between these characteristics and process factors is desired for the industry. This research's focus is investigating feasibility of using optical and scanning electron microscopy to measure powder characteristics and the product properties in LPBF process.

Three different powders were considered for this study as listed below:
1. 316L Stainless Steel is an austenitic stainless steel with chromium (16–18%), nickel (10–12%) and molybdenum (2–3%). It is excellent in elevated temperature tensile, creep and stress-rupture strengths, as well as outstanding formability and weldability
2. Ti-64, Ti-6Al-4V (or Ti 64), is an alloy of titanium with 90% titanium, 6% aluminum, 4% vanadium, 0.25% (max) iron and 0.2% (max) oxygen. It features excellent strength, , high corrosion resistance, good weldability and it is heat treatable.
3. Inconel 718 is a nickel-based super alloy that is well suited for applications requiring high strength in temperature ranges from cryogenic up to 1400°F. Inconel 718 also exhibits excellent tensile and impact strength.
Figure 4 presents three images of Inconel 718 powder with different magnification captured by using Nikon Epiphot 200 microscope and Nikon Ds-L2 camera. (attached file in upload section)
As figure 4 indicates, the optical microscopy has many disadvantageous for this study including limited resolution, small depth of field (focus depth, and losing some features of the particles due to light reflections.
Scanning microscopy on the other hand provides much greater depth of the field and higher magnification. Figure 5 provides images of the same powder with different magnification that are acquired using VSU newly installed SEM.

These images in conjunction with any image processing software can provide a statistical analysis on the size of the particles and size distribution after each production cycle. Due to the images high resolution and detailed features micro defects that are not visible with optical microscopy can be observed.

**Training Opportunities:** Due to some unavoidable circumstances, the SEM installation was delayed. The installation process is under progress and will be completed soon. After the complete installation of the Instrument we will conduct training sessions for both students and faculties. We will record the training session in high definition video and place it on local computers together with copies of the instrument manual to help the researchers to learn how to use the instrument and also to analyze the data obtained from the instrument. After the installation and training of the instrument by the manufacturer, the PI and the engineering lab technician will be responsible for the operation of this instrument. Department of Engineering and Computer Science will provide the technical service and maintenance of the instrument, after the warranty period.

**Results Dissemination:** Nothing to Report

**Honors and Awards:** Nothing to Report

# RPPR Final Report
as of 09-Sep-2019

**Protocol Activity Status:**

**Technology Transfer:** Nothing to Report

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**Person Months Worked:** 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

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Project Contribution:
International Collaboration:
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International Travel:
National Academy Member: N
Other Collaborators:

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**Person Months Worked:** 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

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**Participant:** Xuejun Wen
**Person Months Worked:** 1.00 **Funding Support:**
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International Collaboration:
International Travel:
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Other Collaborators:

**Participant Type:** Other (specify)
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**Person Months Worked:** 1.00

**Funding Support:**

Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

**JEOL 7200 Scanning Electron Microscope at VSU**

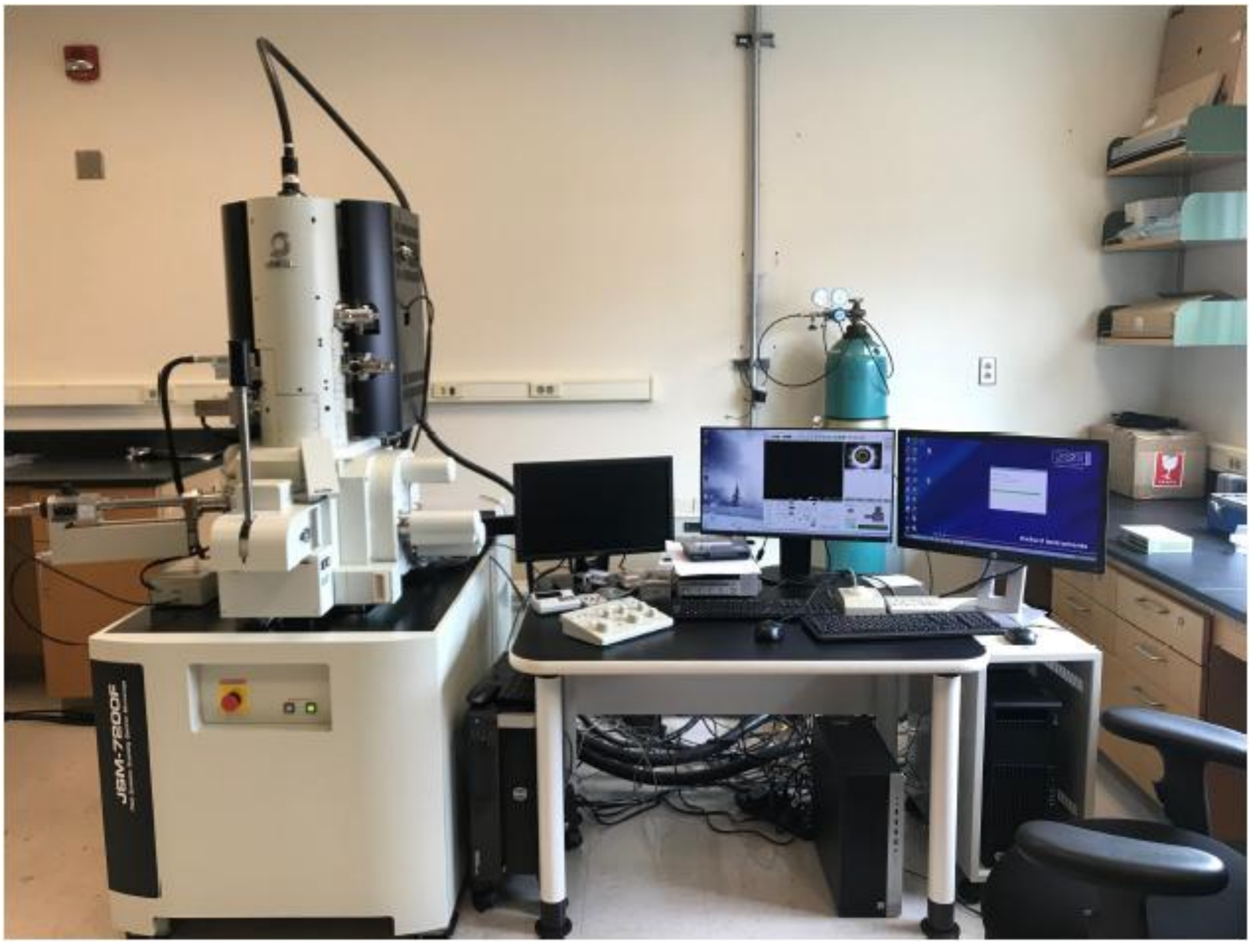


Fig. 1

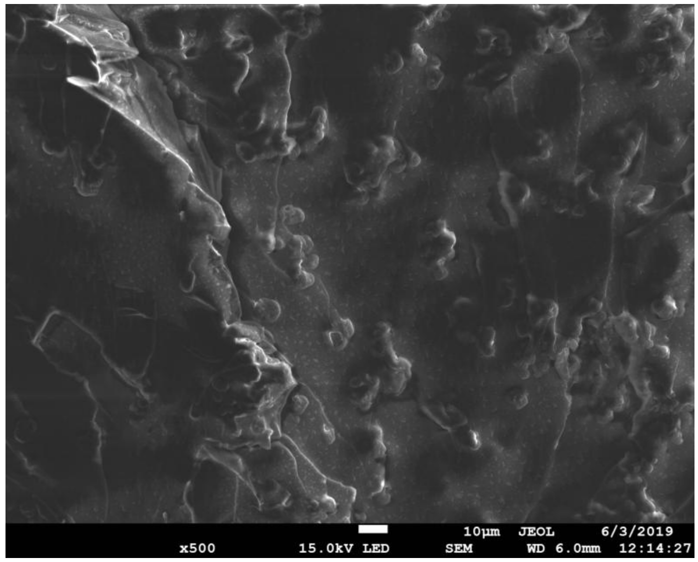



Fig. 2

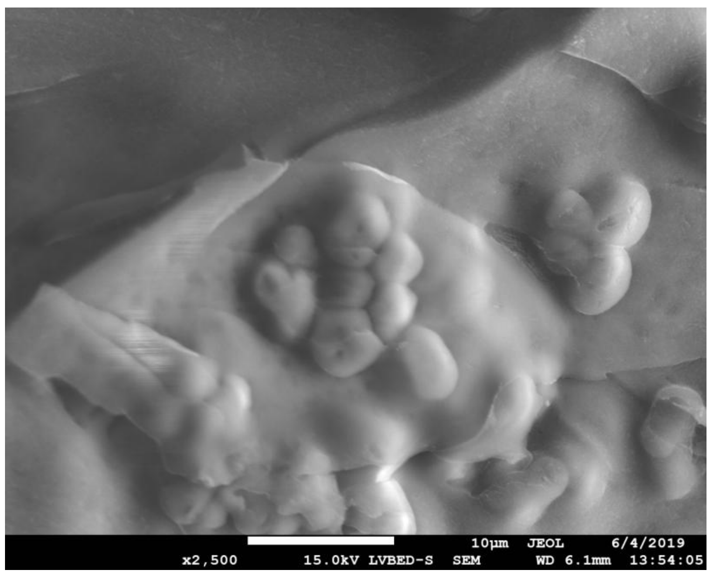
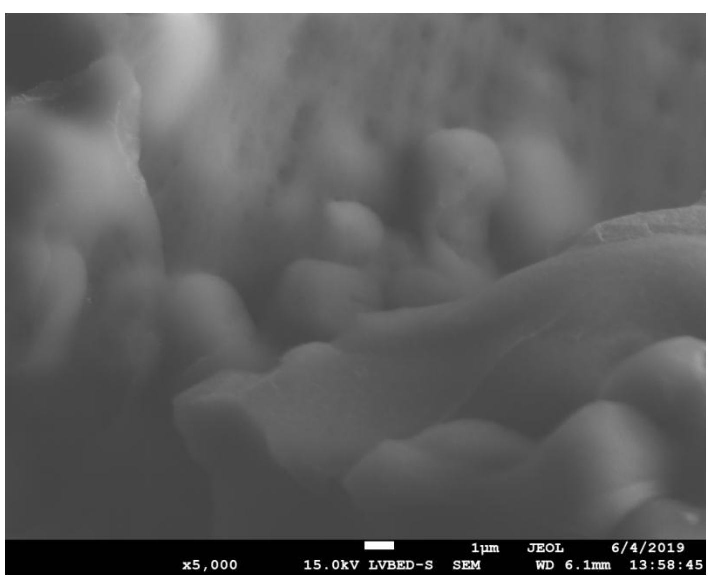



Fig. 3



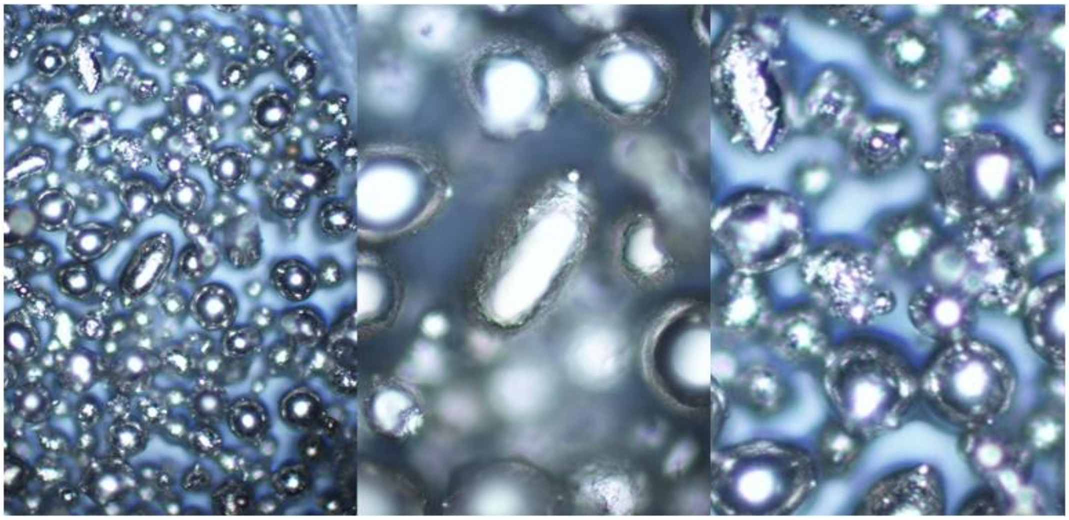


Figure 4 Optical Microscopic Images of the Inconel Powder with different magnification

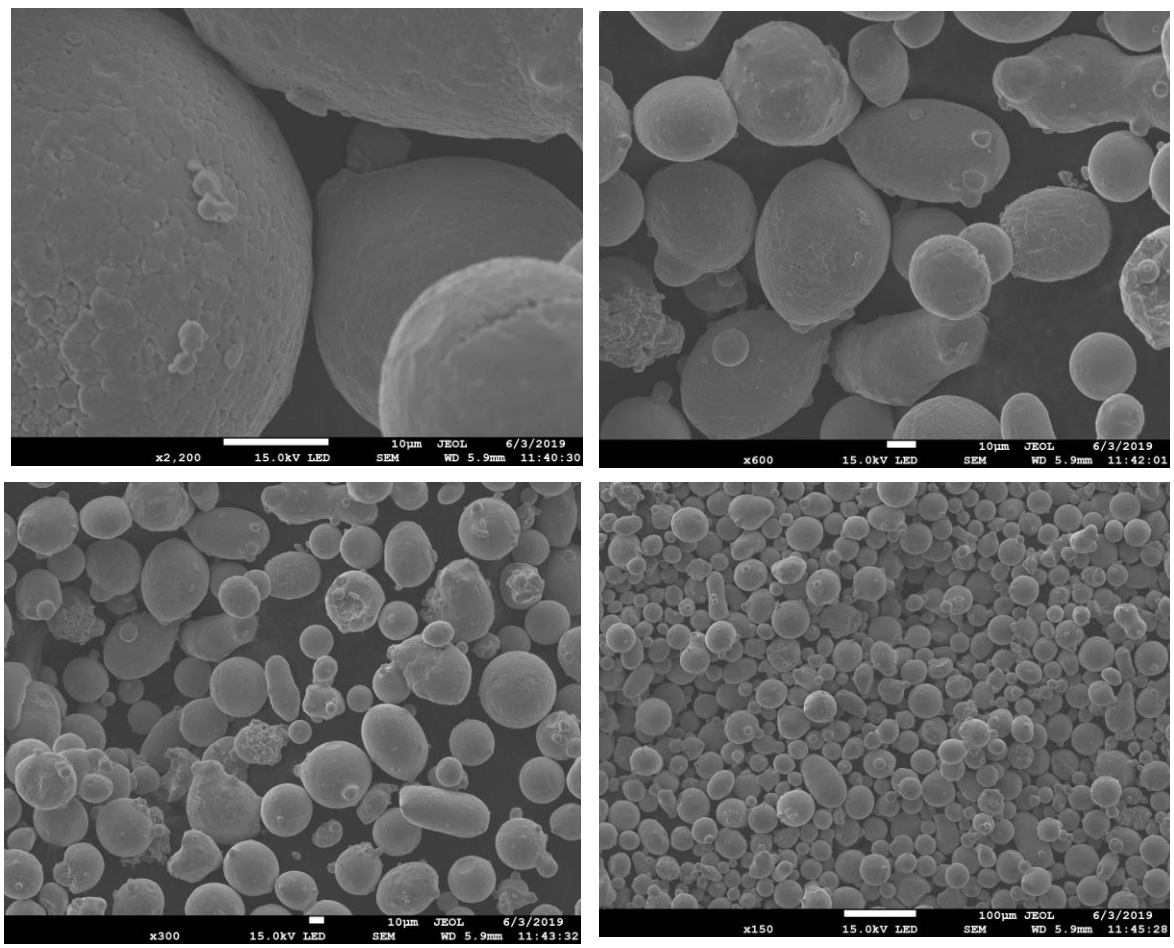



Figure 5 SEM Microscopic Images of the Inconel Powder with different magnification