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Development and Characterization of Aerospace Grade AISI 440C and Cronidur-30 Alloy Powders for Additive Manufacturing Applications

Kumar, M.[1]*, Manojkumar, S.[2], Anto Monish, W.[1], Arunathithyan, N.[1] and Ajay, J.[1]
[1]KPR Institute of Engineering and Technology, Arasur, Coimbatore, INDIA – 641407
[2]National Institute of Technology, Rourkela, Odisha, INDIA – 769008
*Corresponding author (e-mail: kuaymar@gmail.com)

The role of Additive Manufacturing (AM) in high-end engineering industries is expanding rapidly with metal AM becoming a crucial choice, particularly in the aerospace sector. The demand for high-performance, corrosion-resistant metal alloys continues to grow. In line with this, the present study focuses on the development and characterization of aerospace-grade stainless steel alloys (AISI 440C and Cronidur-30), for AM applications, especially in bimetal components. The alloy powders are produced using the gas atomization process with nitrogen inert gas atmosphere and evaluated for their chemical composition, particle size, flowability, apparent density and morphology to ensure their suitability for AM techniques such as Direct Energy Deposition (DED). The chemical composition and microstructure analysis of developed alloy powders are carried out through Inductive Coupled Plasma – Optical Emission Spectroscopy (ICP-OES) and Field Emission Scanning Electron Microscopy (FESEM), respectively.

Keywords: AISI 440C; Cronidur-30; DED; FESEM; XRD; characterization

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High end engineering industries use high performing super alloys in their potential applications like automotive and aerospace. AM processes like DED uses these alloys to produce parts for these applications. There are researches discussing the combined use of Cronidur-30 and AISI440C alloys in bimetallic bearing applications. Cr 30's finer carbides improve rolling contact fatigue (RCF) life by hindering crack propagation, unlike AISI 440C's larger carbides, which promote interconnected cracks and reduce fatigue resistance. Cr-30 achieves optimal hardness and minimal retained austenite within a controlled solutionizing range, reaching a peak hardness of 61 HRC at both 150°C and 475°C. Notably, tempering at 475°C enhances fatigue life by over 0.5-fold compared to 150°C [1]. Another research investigates, residual stresses induced by surface treatments like nitriding have a profound impact on fatigue performance of 32CrMoV13 alloy. Studies demonstrate that compressive residual stresses in near-surface layers can enhance resistance to RCF and RSCF. However, under cyclic loading, these stresses may relax or even reverse to tensile stresses, facilitating crack initiation and propagation [2]. Subsequently, NbC additions into AISI 440C steel leads to a reduction in rod-shaped M_7C_3 carbides at grain boundaries, promoting the formation of spherical M_7C_3 carbides dispersed within the matrix. This transformation enhances the uniformity and toughness of the steel. The optimal transverse rupture strength (TRS) of 1985.2 MPa was observed in specimens with 5 wt% NbC sintered at 1270°C. Higher NbC content (15 wt%) resulted in increased hardness (HRA 80.9) but may compromise toughness due to excessive carbide formation. The same 5 wt% NbC specimens exhibited the highest polarization resistance (1.01×10^2 Ω·cm²), indicating improved corrosion resistance. This enhancement is attributed to the refined carbide distribution and reduced chromium carbide precipitation, preserving more free chromium for passivation [3]. To produce corrosion free alloys, nitrogen content will be enriched in steels under high pressure conditions for the high- end applications. In this approach, nitrogen is introduced during the sintering process, enabling precise control over nitrogen content and distribution, which is beneficial for achieving uniform mechanical properties. Nitrogen is diffused into the steel at elevated temperatures, forming surface-hardened layers that enhance wear and corrosion resistance [4].

There are works that compare the elemental analysis methods viz: ICP-MS and ICP-OES. ICP-MS generally offers superior sensitivity and lower detection limits compared to ICP-OES, making it more suitable for trace element analysis. ICP-OES is effective for analyzing elements at higher concentrations and is less susceptible to certain types of interferences. However, ICP-MS provides a broader elemental range and better resolution, particularly for detecting isotopic variations. Due to its high sensitivity, ICP-MS is preferred for applications requiring the detection of ultra-trace elements, such as in environmental

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monitoring and biological studies. ICP-OES, with its rapid analysis capabilities, is often employed in industrial settings for quality control and materials analysis. In order to customize powder qualities for particular applications, it is essential to optimize the factors. The effectiveness and result of the atomization process are significantly influenced by the metal or alloy's physical and chemical characteristics, such as viscosity, surface tension, and reactivity. Amorphous structures or metastable phases may occur as a result of rapid solidification during atomization, which could affect the powders' mechanical and physical characteristics. For consistent layering and bonding in 3D printing procedures, high-quality spherical powders made by gas atomization are necessary. In coating applications, fine metal powders are utilized to enhance surface qualities like thermal insulation, corrosion protection, and wear resistance [5]. In order to improve efficiency and fine-tune particle dispersion, Lagutkin et al. (2004) introduce a sophisticated metal powder atomization method that combines pressure and gas atomization. In contrast to traditional procedures, the pre-filming step—where molten metal forms a thin film before contact with a gas stream—is highlighted in the study. This results in narrow size distributions and decreased gas usage. With mass median diameters ranging from 20 to 100 µm, experimental results verify the successful atomization of tin and alloy powders, proving the approach's viability for producing high-quality, reasonably priced powders [6].

There is an investigation involved mixing different percentages of TaC powder (10%, 20%, and 30% by mass) with 440C steel powders, followed by an hour of vacuum sintering at temperatures between 1270°C and 1290°C. The findings showed that the strength and hardness of the composites were successfully increased by an ideal amount of TaC. In particular, specimens sintered at 1270°C with a 10% TaC additive showed the highest transverse rupture strength (TRS), measuring 2260.3 MPa. With a hardness of HRA 83.8 following heat treatment, this TRS further increased to 2458.4 MPa. The conversion of rod-shaped M7C3 carbides into M23C6 carbides, which precipitated inside the grains and acted as a strengthening phase, was responsible for the improvement. Conversely, excessive TaC content (30%) led to increased porosity (1.3%), hindering liquid diffusion of iron elements and negatively impacting mechanical properties [7]. Authors have investigated the effects of carbon (C) and nitrogen (N) additions on martensitic stainless steels. Results revealed that, alloying C + N stabilizes the microstructure, resulting in increased hardness, wear resistance, and ductility in comparison to traditional martensitic steels. This study prescribes, Pressurized ElectroSlag Remelting (PESR) is a vital process for enhancing toughness by improving nitrogen solubility and fine-tuning grain structure. The study also reveals how solid-state nitriding can efficiently increase surface hardness, which makes it appropriate for applications that need high wear and fatigue resistance. Results prove the potential of martensitic stainless steels enriched with C and N for sophisticated technical uses such as manufacturing systems, industrial and aerospace applications [8]. With this profound literature exposure, the present study aims to develop the novel aerospace grade AISI 440C and Cronidur 30 alloy powders through gas atomization process and characterize them.

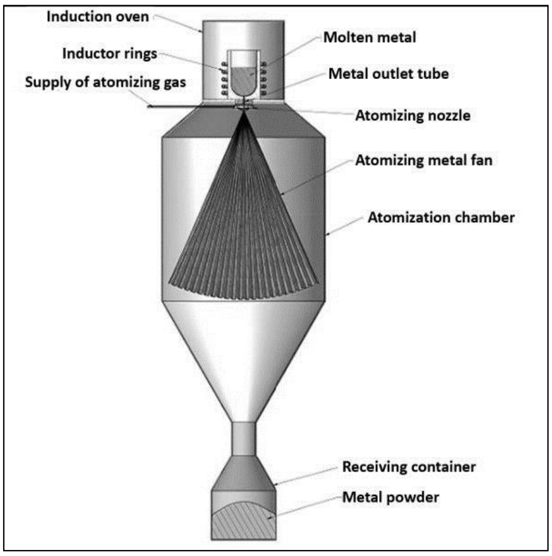

Figure 1. Gas atomization process setup [9].

EXPERIMENTAL METHODS

This section consists of the methods of production and characterization of AISI 440C and Cronidur 30 alloy powders.

Gas Atomization Process

The technique of gas atomization (Figure 1) uses a high-pressure gas stream to quickly cool molten metal in order to create fine metal powders. This method breaks down molten metal into tiny droplets by exposing it to a high-velocity gas, such as nitrogen, after it has been ejected via a nozzle. These droplets rapidly cool and solidify to form uniformly tiny powder particles. Because of their high purity and regulated particle size, the resultant metal powders find use in a variety of industries, such as coating applications, additive manufacturing, and powder metallurgy.

Development of AISI 440C and Cronidur 30 Alloy Powders

A martensitic stainless steel (AISI 440C) widely used for its excellent hardness, wear resistance, and moderate corrosion resistance, involves several critical steps to ensure high-quality properties. Initially, fine powders of AISI 440C are produced through atomization techniques, ensuring a controlled particle size and shape suitable for AM processes. Cronidur-30, a high-performance alloy known for its exceptional wear and corrosion resistance [10]. The process begins by melting the respective alloys in a controlled atmosphere or vacuum induction furnace to ensure uniform composition and prevent contamination or oxidation. The molten metal is then discharged through a nozzle into an atomization chamber, where it is subjected to high-pressure nitrogen inert gas, introduced through strategically designed jets. The high-velocity gas stream atomizes the molten metal into fine droplets, which rapidly solidify into spherical particles as they travel through the chamber. This rapid cooling minimizes segregation and porosity, yielding powders with consistent properties and excellent suitability for advanced applications. The as fabricated powders are shown in Figure 2.

The solidified powders are collected at the bottom of the chamber or using a cyclone separator, followed by screening and classification to obtain the desired particle size distribution tailored to additive manufacturing applications. Post-processing steps, including annealing, passivation, or coating, are employed to enhance flowability, reduce surface oxidation, and improve powder stability. Rigorous quality control tests, such as particle size analysis, flowability measurements, and chemical composition validation, ensure the final powders meet stringent industry standards. The gas atomization process produces AISI 440C and Cronidur-30 powders with excellent sphericity, high purity, and superior performance attributes, making them indispensable for AM applications. The AMed products require appropriate heat treatment before it is getting applied for industrial or commercial needs. Research results demonstrate that thermo-mechanical treatment (TMT) leading to uniform carbide dispersion and improved hardness above 58 HRC, enhancing wear resistance and fatigue life in AISI 440C steel. Also, heat treatment effects on N_2 containing martensitic stainless steel, showing that Mo_2C and Cr_2N precipitates formed at 500°C significantly improve hardness (446 HV to 620 HV) and fatigue performance of 17Cr-0.17N-0.43C-1.7 Mo Martensitic Stainless Steel [12, 13].

Figure 2. As developed Cronidur 30 and AISI 440C alloy powders.

Table 1. Chemical composition of AISI 440C and Cronidur-30 alloy powder particles.

Elements	AISI 440C (wt.%)	Cronidur-30 (wt.%)
Cr	17.5	15.3
Si	0.56	0.90
Mo	0.20	0.96
Mn	0.90	0.89
Ni	0.20	0.12
C	1.10	0.34
S	<0.003	<0.002
N	-	0.39
Fe	balance	balance

RESULTS AND DISCUSSION

Powder Characterization

Powder characterization such as particle size, shape, apparent density, and composition affect processes like compaction and sintering. Techniques like ICP-OES, XRD, FESEM and EDS provide comprehensive analysis, are used in combination for a complete understanding of powder behavior, and are detailed in the upcoming sections.

Chemical Composition Analysis

The Table 1 shows chemical composition analysis of the gas atomized AISI 440C and Cronidur-30 alloy powder particles which are performed using Inductive Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). From the results it is observed the alloying substances in these two types of alloys are very similar. However, Cronidur-30 alloy has doped with small quantity of nitrogen which is absent in the AISI 440C alloy powder particles.

Particle Size Analysis

Figure 3 and 4 shows the detailed analysis of the particle size of the gas atomized AISI 440C and Cronidur-30 alloys powder particles. The AISI 440C material exhibits particle size distribution, in which 3.69% of the particles surpass 90 µm, and 20.62% are within the 90-75 µm range. The predominant particles range from 75 to 45 µm, constituting the balanced fraction, and 6.25% of the particles are less than 45 µm.

Cronidur-30 exhibits a distinct particle size distribution, with 5.37% of particles beyond 90 µm and 20.99% falling within the 90-75 µm range. The major volume percentage is located between the 75-45 µm range, while 5.57% of particles are less than 45 µm. The particle distribution analysis shows that the AISI 440C has little finer particles however overall mean particle size distribution is almost similar.

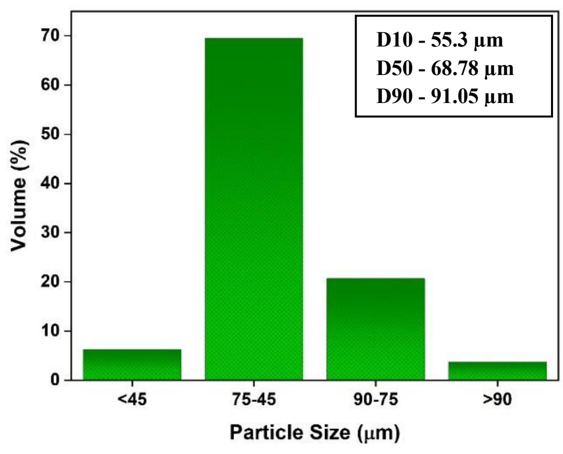

Figure 3. Particle size (diameter) analysis of AISI 440C alloy powder.

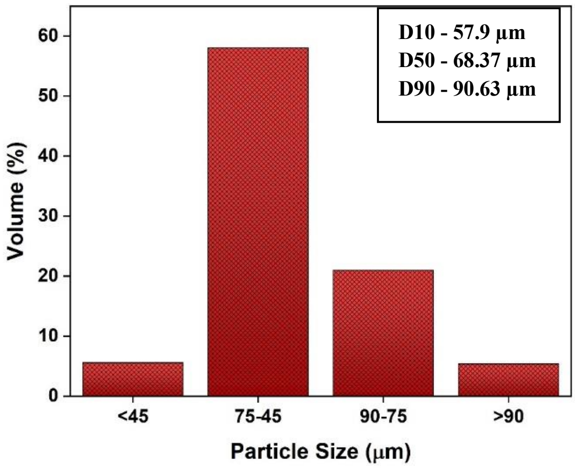

Figure 4. Particle size (diameter) analysis of Cronidur-30 alloy powder.

In the insert of Figure 3 and 4, the D10, D50, and D90 of powders representing the particle diameters (size) below which percentage of all particles are observed. According to the results, the powders of AISI alloys are found to possess a size distribution of more than 90% (D90) particles that are less than 91.05 μm, 50% of the powder particles are smaller than 68.78 μm, and 10% of the powder particles are smaller than 55.3 μm. Similarly, Cronidur-30 alloys powder having particles size of D90 - 90.63 μm, D50 - 68.37 μm and D10 - 57.9 μm respectively.

Physical Properties of Alloy Powders

Hall Flow Test: The hall flow rate of the AISI 440C alloy powder is measured as18.32 s per 50 g, indicating that 50 g of AISI alloy powders passed through a Hall flowmeter funnel in 18.32 s. On other hand, Cronidur-30 had a flow time of 19.19 s per 50 grams, which indicates that it has a marginally decreased flowability (Table 2). These tests are conducted as per ASTM B212 standard.

Apparent Density Test: The powder particles' apparent density is the mass of a unit volume of loose powder, measured in grams per cubic centimeter. The AISI material possesses an apparent density of 4.05 g/cc, whereas Cronidur-30 displays a little enhanced density of 4.12 g/cc (Table 2). Slightly lower apparent density in the AISI 440C alloy powders may be the more irregular and high surface roughness as compared with the Cronidur-30 alloy powders. ASTM B213 standard is followed to perform this test.

X-Ray Diffraction (XRD) Analysis

The X-ray Diffraction (XRD) patterns of AISI 440C and Cronidur-30 alloy powders were illustrated in Figure 5 and Figure 6 respectively. The XRD pattern shows the peaks the 2θ peaks at 43.39, 44.23, 50.51 and 74.20. The XRD analysis confirms that the peaks at 43.39, 50.51 and 74.20 are belongs to the austenite phase. The peak at 44.23 degree corresponds to the $Cr_{23}C_6$ (Metallic phase (MC)) phase. The presence of the MC phase is the one of major constituent phase which will help in strengthening AISI 440C alloy in the subsequent processing like sintering or additive manufacturing.

Table 2. Physical properties of AISI 440C and Cronidur-30 alloy powder particles.

Alloy	Flow rate (s/50 g)	Apparent density (g/cc)
AISI 440C	18.32	4.05
Cronidur-30	19.19	4.12

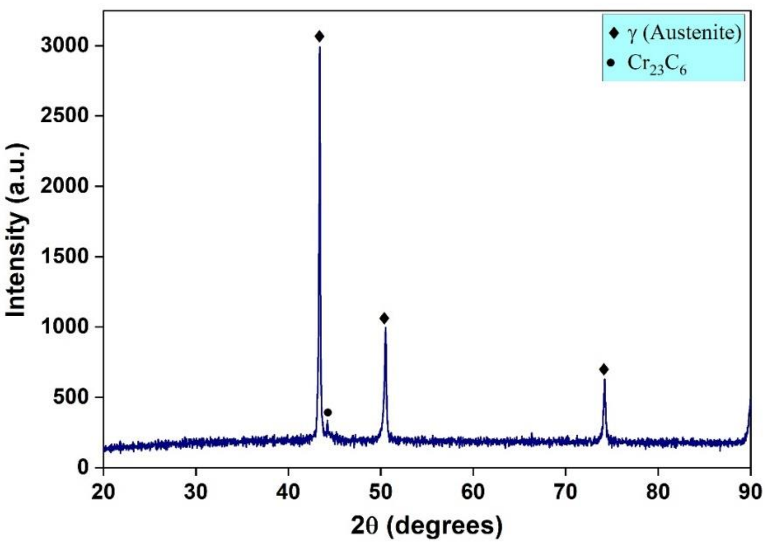

Figure 5. XRD pattern of the AISI 440C alloy powders.

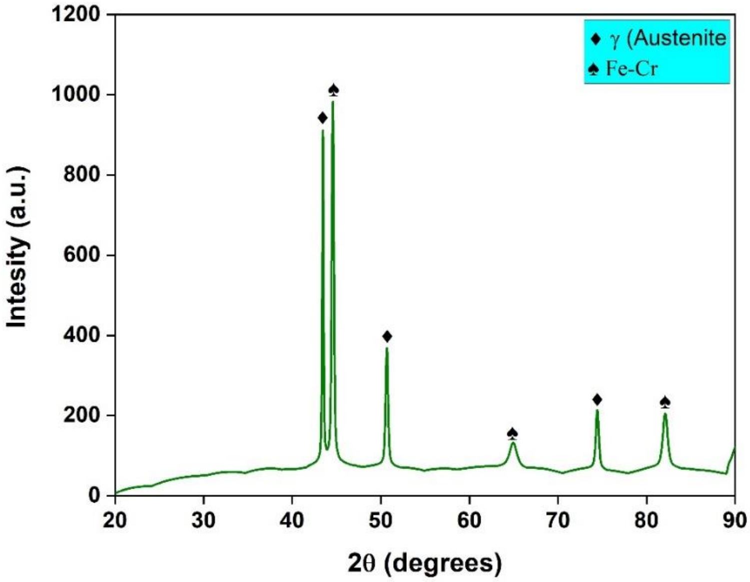

Figure 6. XRD pattern of the Cronidur-30 alloy powders.

The XRD pattern of the Cronidur-30 alloys powders shows peaks 2θ at 43.46, 44.57, 50.57, 64.93, 74.43, and 82.09. Cronidur-30 alloys also have presence of austenite phase at 43.46, 50.57, and 74.43 degrees. In addition to that gas atomized Cronidur-30 alloy powders displays the high intensity of Fe-Cr solid solution cubic phase at 2θ at 44.57, 64.93, and 82.09 degrees.

FESEM Analysis

The FESEM micrographs (Figure 7 and 8) of AISI 440C and Cronidur-30 powder particles exhibited a uniform spherical shape, medium size and showing almost a similar structure. The spherical shape morphology of the powder particles is the characteristic feature of the gas atomization process. Spherical shape morphology is achieved in the both alloy powder particles which indicates that the alloy powders are perfect for additive manufacturing techniques like direct energy deposition and powder bed fusion. The spherical shape particle surfaces are very smooth which has good flow ability as compared to the irregular particles. In both the alloys powder particles display the existence of some small particles stick over larges particles. This phenomenon is called as satellite (Encircled in the Figure 7 and 8).

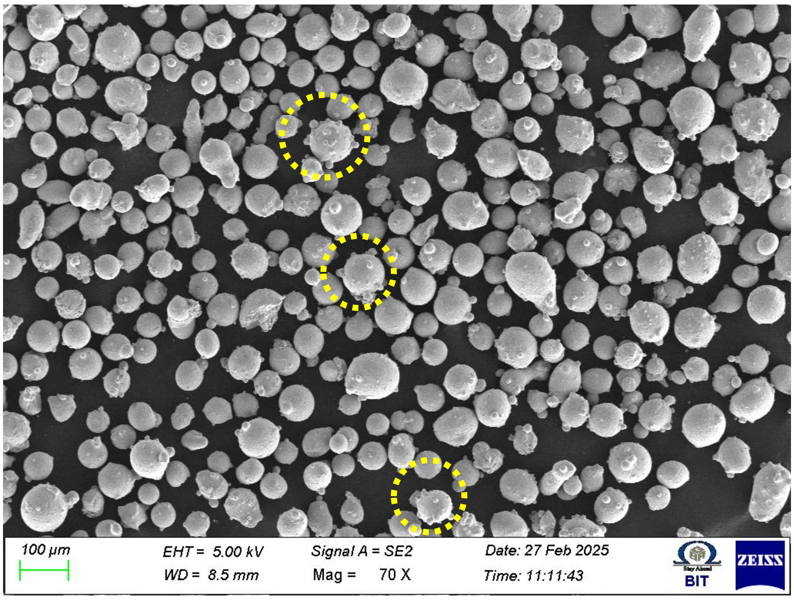

Figure 7. FESEM micrograph of the AISI 440C alloy powder.

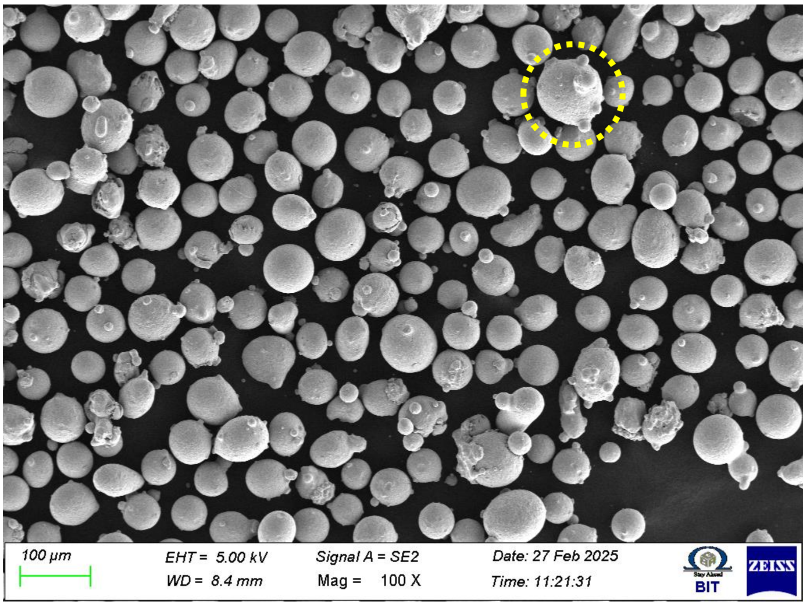

Figure 8. FESEM micrograph of Cronidur- 30 alloy powder.

This satellite phenomenon is generated, as a result of fine powder adhering to coarse powder during gas atomization process. Generation of these satellite structure will affect the packing density and results in formation of defects. These satellite particles also affect the flowability of the powder particles as a negative side. Also, since the surface area of the powders is increased it may induce oxidation/ contamination. The Cronidur-30 alloy powder particles have little high in size prone to stick on the surface of the regular spherical morphological particles as compared with the AISI 440C, because of that Cronidur-30 alloy powders had little high flow time as compared with the later alloy powders during hall flow testing.

CONCLUSIONS

The present research work successfully developed aerospace-grade AISI 440C and Cronidur-30 stainless steel powders through gas atomization process for additive manufacturing applications. Following are the results drawn from various characterization tests conducted.

- Chemical composition analysis carried out via ICP-OES has revealed that Cronidur-30 alloy has doped with significant quantity of nitrogen which is absent in the AISI 440C alloy.

- Particle size analysis report reveals that AISI alloys are found to possess a size distribution of more than 90% (D90) particles that are less than 91.05 µm, 50% (D50) of the powder particles are smaller than 68.78 µm, and 10% (D10) of the powder particles are smaller than 55.3 µm. Similarly, Cronidur-30 alloys powder having particles size of D90 - 90.63 µm, D50 - 68.37 µm and D10 - 57.9 µm respectively.

- Cronidur-30 has recorded a marginally decreased flowability compared to AISI 440C alloy powder as per Hall flow test.

- The AISI 440C possesses an apparent density of 4.05 g/cc, whereas Cronidur-30 records a little higher density of 4.12 g/cc may be due to more irregularity and high surface roughness.

- The XRD analysis confirms the presence of the austenite phase in AISI 440C alloy along with MC ($Cr_{23}C_6$) phase which strengthens the alloy. Cronidur-30 contains austenite with solid solution Fe-Cr cubic phase.

- The FESEM micrographs of AISI 440C and Cronidur-30 powder particles exhibited a uniform spherical shape, medium size and showing almost a similar structure, and satellite phenomena.

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