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Powder Metallurgy HIP for Naval Nuclear Applications – Trends in Process and Property Development

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Abstract. Powder Metallurgy - Hot Isostatic Pressing (PM-HIP) is considered a key technology for component fabrication. By offering near-net shape forming, long lead-time components can be delivered quicker and more efficiently, ultimately supporting on-time construction of nuclear components and structures. To this end, ferrous (A508 Grade 4N) and nickel-base alloys (A600) have been examined in the consolidated PM-HIP condition to assess the mechanical behavior as well as similarity to their wrought/forged counterparts. In this study, various aspects of the PM-HIP process were explored from the powder production to the consolidated material and eventual heat treatment to develop a greater understanding of optimized mechanical properties of PM-HIP material. Trends in processing conditions and various heat treatments were correlated to the performance of each material and related to the wrought counterpart.

Introduction

The United States Navy recently issued its 30-year shipbuilding plan which outlined the number of vessels and platforms to be acquired each year to meet US Navy force-level goals [1,2]. As part of this document, it was identified that in order to meet the future demands of naval nuclear vessel construction (nuclear submarines and aircraft carriers) shipyard output for submarines alone will need to increase by ~250% over the next ~10 years compared to the previous ~10 years. This includes production of the future COLUMBIA-class of ballistic missile submarines which have been identified as the Navy's #1 priority. To meet this demand, the Naval Nuclear Laboratory (NNL) and shipyards have begun to aggressively pursue advanced manufacturing technologies that assure targeted production goals can be achieved and offer the potential to alleviate strain on the current vendor base. PM-HIP has been identified as one such technology and has the potential to displace and/or compliment the current production capacity for naval nuclear components. Apart from enabling manufacturing of critical naval nuclear components, PM-HIP also allows for potential performance/quality improvements and cost/lead time reductions over legacy processes such as casting and forging.

Materials

Materials under consideration for PM-HIP applications include both ferrous and nickel-base alloys, however, the remainder of the discussion will focus on ASTM A508 Grade 4N Class 1 (A508Gr4N), low alloy steel. Two chemistry variants of A508Gr4N, conventional and low residuals, were examined in this study. Powder was sourced from three different vendors who produced the powder using either vacuum inert gas atomization (VIGA) or inert gas atomization (IGA). Powder details are shown in Table 1. It was determined that the IGA process could only achieve conventional chemistries but was still included for powder and mechanical property comparisons.

Table 1 – Powder and HIP details for A508 Grade 4N material analyzed.

Vendor	Target Composition Type[1]	Melt Method	Atomization Gas	Nominal PSD [µm]	Powder Oxygen Content (ppm)	Billet Outgas Temperature	Billet HIP Temperature / Time
A	Low Residuals	VIGA	Nitrogen	0-500	200	70°F, 250°F, 500°F	2065°F – 4 hrs or 2215°F – 4 hrs
	Low Residuals	VIGA	Nitrogen	53-500	140		
B	Low Residuals	VIGA	Nitrogen	53-500	150	70°F, 250°F	2190 – 6 hrs
C	Conventional	IGA + No Cover Gas	Nitrogen	50-150	190	400°F	2250°F – 4 hrs

[1]Low residuals contain low levels of Si, Mn, P and S compared to conventional compositions.

Upon receipt of the material from each vendor, the powder was analyzed via Scanning Electron Microscopy (SEM) and Auger electron Spectroscopy (AES) to understand basic powder characteristics and to characterize the inherent oxide layer on the surface of all particles. Of the two lots of powder from Vendor A, 12 billets (~17"x8"x8") were HIP consolidated (6 from each PSD) with each undergoing a unique processing sequence. This allowed any variation in properties due to particle size distribution (PSD), outgas temperature, and HIP temperature to be uniquely defined. Remaining powder from Vendors B and C was HIP'd under the parameters shown in Table 1 to form billets of 22"x8.5"x8.5" and ~20"x9"x5" respectively.

Following HIP consolidation of all 15 billets, a standard austenization (1575°F – 4 hrs), water quench and temper (1205°F +/-25°F – 10 hrs) was applied to billet sub-sections measuring ~8"x4"x4" with the canister still present. Billet sub-sections were then further sectioned into tensile and Charpy specimens. Tensile specimens were tested at 70°F (per ASTM E8), while Charpy specimens were tested at a range of temperatures (per ASTM E23) to define the ductile to brittle (DTB) transition associated with PM-HIP A508Gr4N.

Results and Discussion

Powder Analysis – Oxide Thickness Correlation

Manufacturing A508Gr4N powder is relatively new to the existing vendor base, and it was desired to characterize the current powder production capabilities. The VIGA and IGA powders obtained from the three vendors was first analyzed under the SEM with images of each powder shown in Fig. 1. Much of the powder produced via the VIGA process was characterized as having spherical or oblong particles with moderate to heavy satellite particles attached to the main particle. Conversely, the IGA powder particles exhibited a more ideal particle geometry consisting of spherical particles with smoother particles surfaces and fewer satellites.

After examining the powder morphologies, each powder was analyzed using AES to characterize the particle and satellite oxide thickness from powder produced at each vendor. AES clarified that there was not a significant variation in the oxide thickness from the VIGA and IGA processes. However, depth profiles indicated that thinner oxides were consistently observed on powder that was spherical in morphology and had fewer satellites, whereas particles with mottled surfaces and increased satellites had comparatively larger oxide thicknesses as shown in Fig. 2. This would suggest that the powder production method has limited bearing on the ability to produce powder with a quality morphology as powder produced using the IGA process possessed the most favorable powder particle morphology and fewest satellites. Variation in powder morphology may be correlated to vendor experience with particular alloys, as Vendor C is known

to regularly produce AISI 4340, while Vendors A & B had limited experience producing low alloy steel powder.

When the two PSD as produced from Vendor A were examined, the oxide thickness was found to be consistent between the full cut of powder and larger PSD. Thus, the oxide thickness on all has no correlation to particle size. This corelates with the best practice to screen out the small particles or "fines" due to the increased surface area corresponding with elevated oxygen content that will carry into the HIP consolidated component.

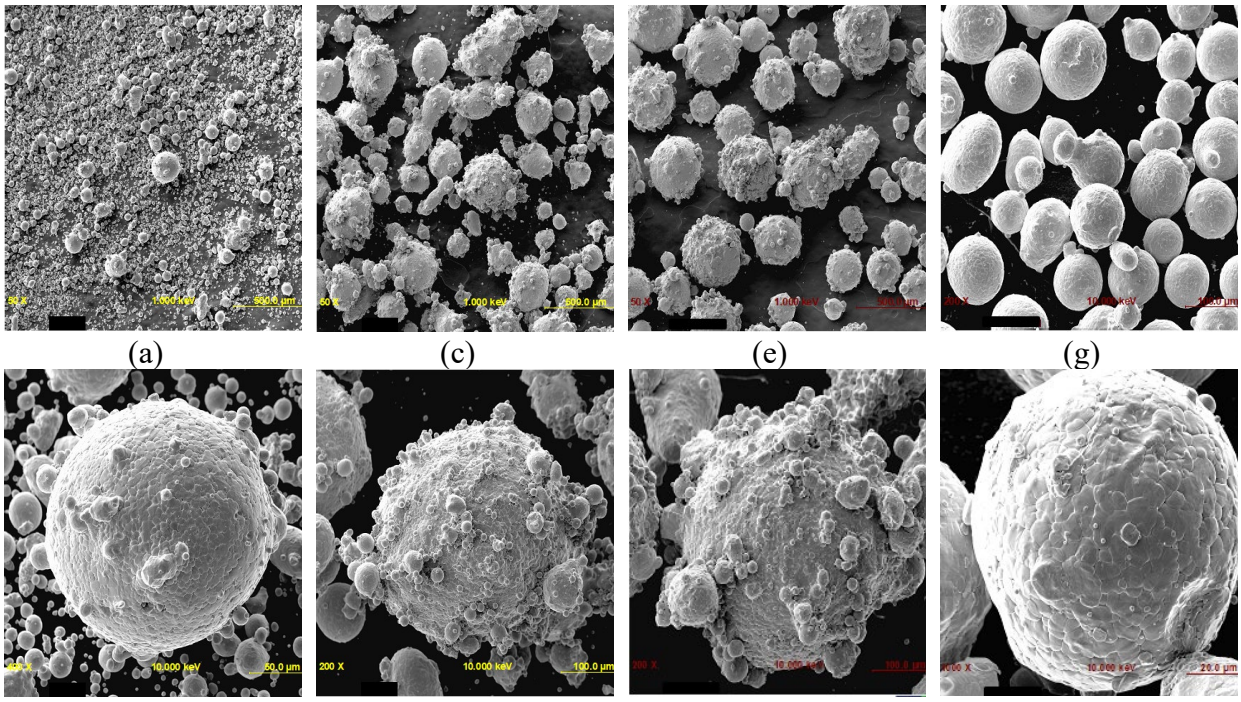


Figure 1. Powder Particles from (a-b) Vendor A – VIGA, full cut powder, (c-d) Vendor A – VIGA, fines removed, (e-f) Vendor B – VIGA, fines removed, and (g-h) Vendor C – IGA, fines removed.

Mechanical Properties of HIP Consolidated Material

Consolidated and heat treated PM-HIP material was mechanically tested to compare PM-HIP properties to the wrought ASTM A508Gr4N equivalent as well as identify the DTB transition curve. Tensile and Charpy specimens were extracted from billet sub-sections in a grid-like pattern with a general extraction location detailed in Fig. 5.

Tensile properties for all billets from all three vendors were found to have room temperature tensile properties that exceeded the minimum requirements for ASTM A508 Grade 4N Class 1 with no single data point falling below the minimum requirements. Tensile properties among the three Vendors had little deviation in strength while maintaining considerable ductility despite the variation in melting/atomization practice and resulting differences in chemistry (conventional vs. low residual). This suggests that the PM-HIP process may be robust enough to recover from slight variations in supplier chemistry, or powder production method, if only tensile properties are required. The robust nature of the tensile properties are highlighted in Fig. 3 which depicts 12 unique processing conditions for Vendor A and shows minimal variation for each condition. Further testing is recommended to confirm this trend, however, the limited variation suggests that hot outgassing or screening out smaller particles is not required to achieve minimum required tensile properties. This has the potential to save costs and reduce production span time.

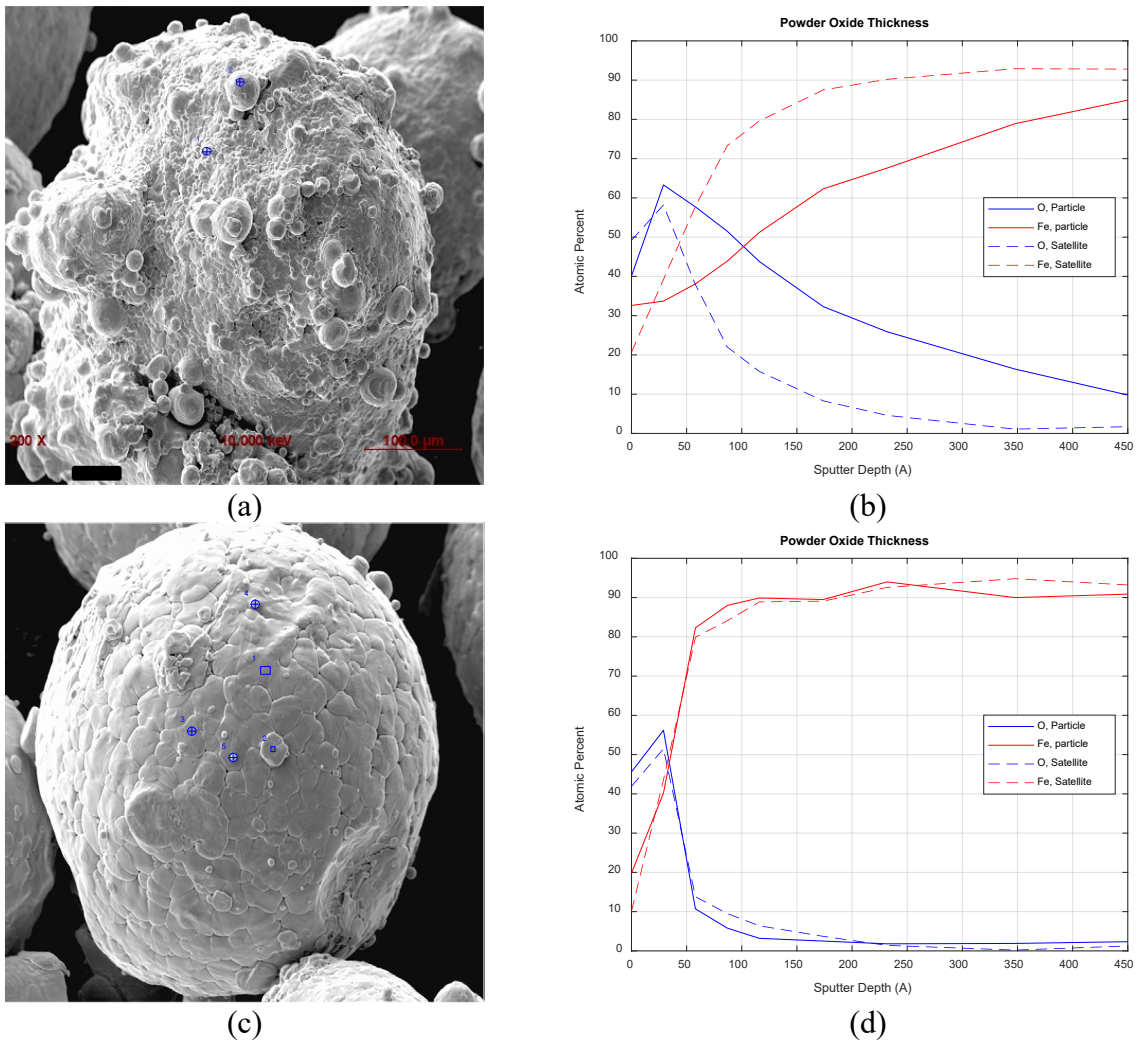
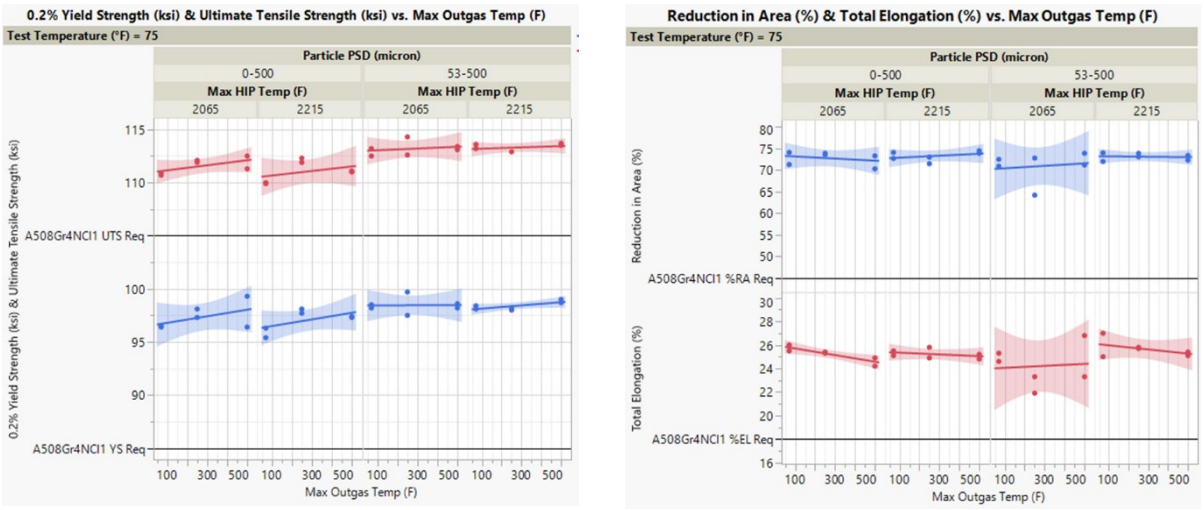


Figure 2. Powder oxide thickness plots for representative (a-b) VIGA produced powder, Vendor A & B and (c-d) IGA Produced powder, Vendor C.

Near Canister Degraded Impact Toughness

The Charpy impact toughness response for each of the billets is shown in Fig. 4. Analysis of the impact data provides two key conclusions (1) there is a significant reduction in properties for the IGA produced powder compared to the VIGA powder and (2) there is a large amount of scatter in the existing data. The reduced impact toughness of the IGA powder is highlighted in Vendor C possessing significantly lower impact toughness which consistently measured more than 50 ft-lbs below the VIGA produced material from Vendor A. The ASTM required average impact toughness for A508Gr4N is 35 ft-lbs at -20°F with no specimens measuring below 30 ft-lbs. Powders from all three vendors exceeded this requirement. Only powder from Vendor A exceeds a notional 100ft-lb target the Electrical Power Research Institute (EPRI) has established for PM-HIP A508 Grade 3 development [3].



(a) (b)

Figure 3. Tensile properties for unique processing conditions of Vendor A powder (a) strength and (b) ductility.

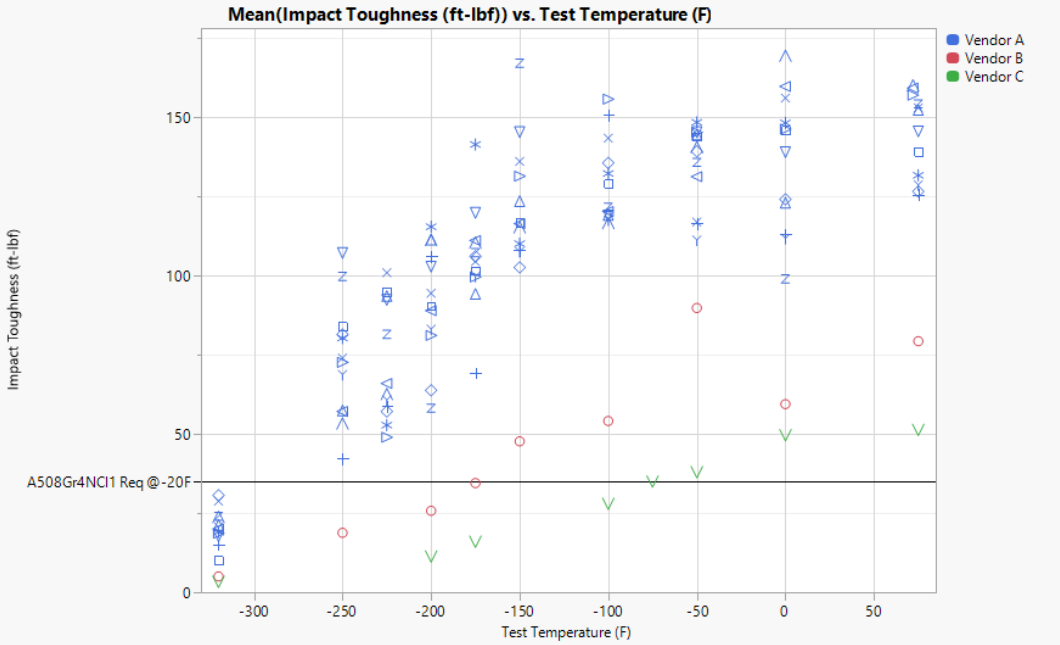


Figure 4. Charpy impact toughness data from each of the three vendors.

The degree of scatter observed in Fig. 4 was not expected and resulted in further analysis of individual data points for each of the billets tested. Conveniently, the grid-like pattern used for Charpy specimen in Fig. 5a allowed for pseudo heat maps to be developed to determine location specific toughness across each billet. Fig. 5b shows up to 100 ft-lbs variation in impact toughness when comparing the specimens located immediately adjacent the original canister (specimens 10-17 in Fig. 5a) compared to the specimens located in the billet interior. By separating the location specific toughness response, the scatter is diminished, and the PM-HIP material exhibits two unique DBT curves which was observed across all vendors and powder types. The specimens extracted from the billet interior were found to have improved impact toughness compared to the near-can region with upper shelf toughness for Vendor A interior billet specimens ranging from 140-175 ft-lbs compared to ~90 ft-lbs for near can regions.

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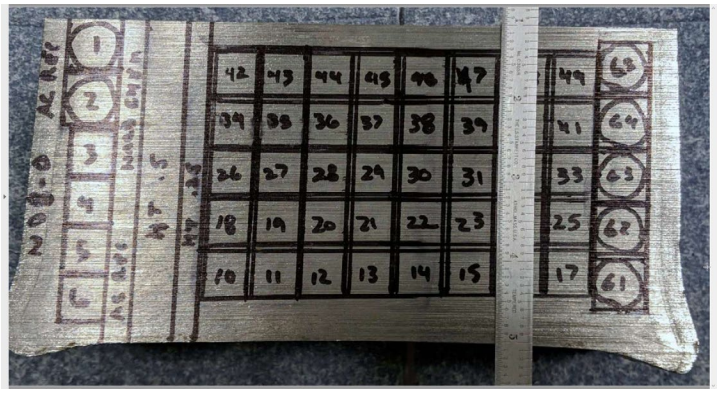
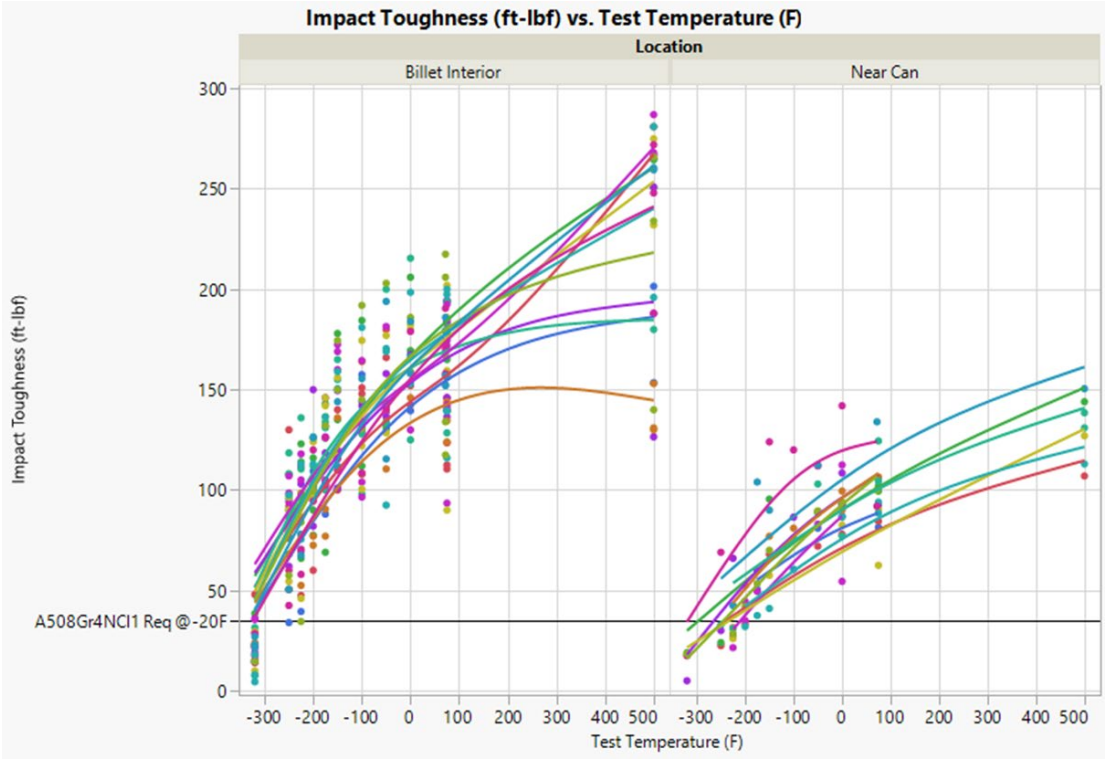


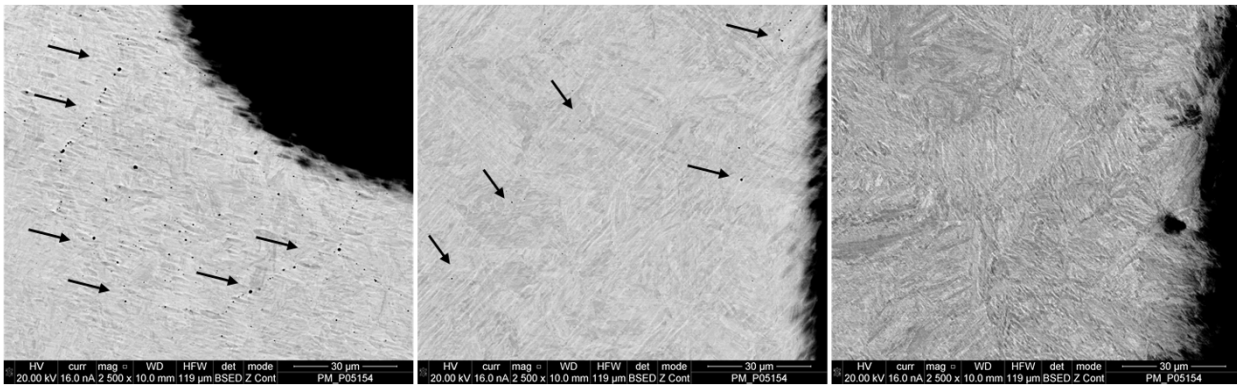
(a)



(b)

Figure 5. (a) Charpy and tensile specimen extraction location and (b) DTB Transition curve highlighting the near can response for billets from Vendor A.

The mechanism resulting in the degraded properties in the near-can region are currently not well understood. Fractography of the Charpy specimens fracture surfaces exhibited ductile tearing or microvoid coalescence for both locations and no distinct variation. Kinetic or chemical effects were also ruled out as both μXRF and electron probe microanalysis scans across the canister-billet interface showed no chemical variation. The only variation between the two regions of the PM-HIP billets is the presence of increased oxide inclusions decorating prior particle boundaries in the near-can specimens compared to the billet interior specimens as shown in Fig. 6. The increased number of inclusions creates a network which may result in a preferential cracking pathway and reduced toughness in material up to1-1.5" into the billet interior as measured from the original canister surface. Note that all tensile specimens were extracted from the "near-can" region but still exhibited acceptable properties.



(a) (b) (c)

Figure 6. SEM BSE images of near-notch Charpy specimens showing inclusions indicated by black arrows (a) along prior particle boundaries of a near-can specimen (b) scattered in interior billet specimens and (c) forged A508Gr4N microstructure for comparison.

Conclusion

NNL examined consolidated PM-HIP A508Gr4N billets fabricated with both VIGA and IGA powder. The tensile properties of the VIGA and IGA material was found to be equivalent to typical wrought tensile properties despite all tensile specimens being extracted from near canister locations. The Charpy results showed significant variation between Vendor A B and C. All material exceeded the current ASTM A508 Grade 4N Charpy impact requirements, but only Vendor A exceeded a notional 100ft-lb target value at room temperature. General property evaluation suggests that even though quality particles are obtainable via the IGA process, the Charpy impact properties are degraded relative to VIGA powder. Despite different melting and outgas/fill method, all 15 billets examined within this study exhibited a reduction in properties near the canister and was estimated to be ~1-1.5" into the material as measured from the canister. It appears that there may be an increased level of oxides at prior particle boundaries in these regions though results are not conclusive. The mechanism for increased oxide content near the canister is not clear at this time and requires additional investigation.

References

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