Characterizing the Geometry & Dispersity of TiN Inclusions in Carpenter Technology® Custom 465® Stainless Steel

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Abstract: Carpenter Technology® is seeking to characterize the formation of titanium nitride (TiN) inclusions during the solidification of specialty alloy Custom 465®. Several samples were taken from a Custom 465 VIM ingot, and an optical analysis method for identification and statistical analysis of TiN was used to characterize inclusions within the material. From this analysis, statistical data relating to the particle area and inclusion area fraction were determined and compared to literature values. In addition, an equilibrium solidification simulation was run in Thermo-Calc® 2023a to determine the volume fraction of TiN in Custom 465.

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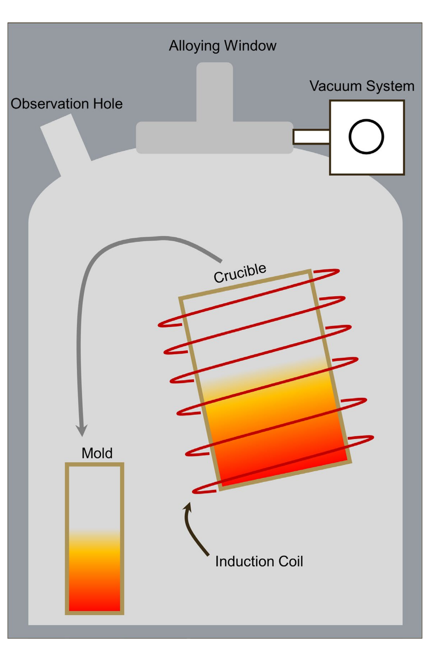
CARPENTER® TECHNOLOGY

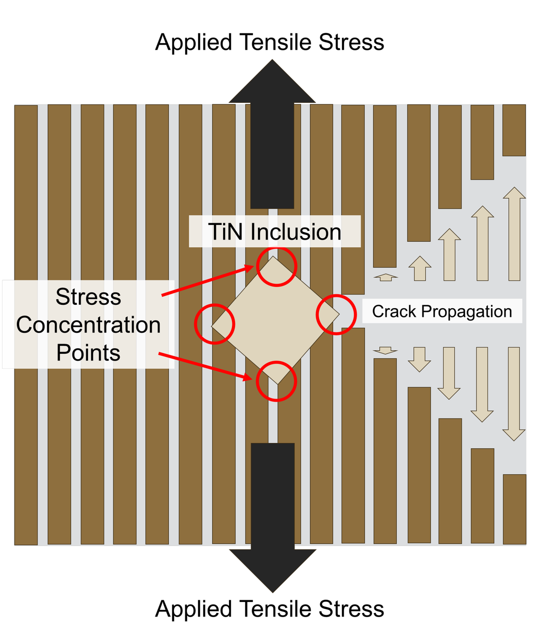
Background

Custom 465® is a high-strength stainless steel used in aerospace applications that require a combination of strength, toughness and corrosion resistance such as landing gears. It is manufactured by Vacuum Induction Melting (VIM) to minimize the formation of oxides followed by Vacuum Arc Remelting (VAR), a secondary remelting process by which a VIM ingot is further cleaned, and solidified grain development and microstructure are further controlled.

Element	Cr	Ni	Mo	Ti	Fe
Wt. %	11-12.5	10.8-11.3	0.8-1.2	1.5-1.8	Balance

[1]

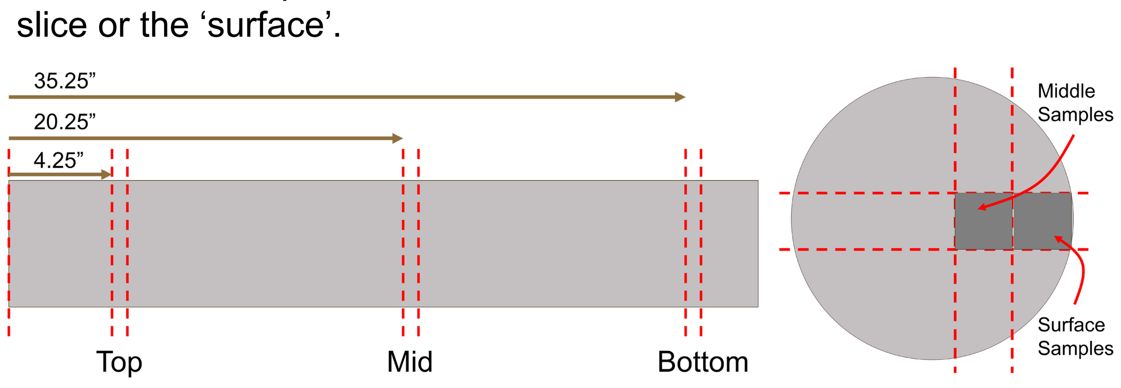




Because it is not possible to completely remove Nitrogen (N) from the melt, Titanium Nitride (TiN) inclusions naturally occur in Ti-containing steels and weaken the metal notably. The particles break up homogeneity in the matrix and the harsh edges create locations for easy crack formation and propagation [1-2]. Since the formation of these particles cannot be avoided, Carpenter Technology® has tasked us with characterizing their formation and distribution in the ingot during VIM. To achieve this, our goal was to analyze the surfaces of Custom 465 stainless steel ingots as well as to create complex computer models capable of tracking TiN formation and trajectory in the melt during VAR melting and solidification.

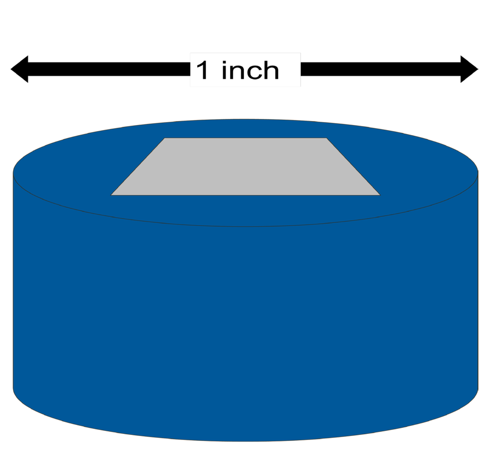
Experimental Methods

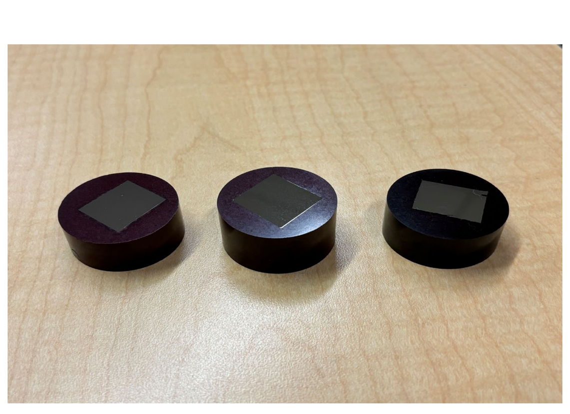
Thirteen samples of Custom 465 stainless steel produced via Vacuum Induction Melting (VIM) were supplied by Carpenter Technology. These samples were removed from a ¾ inch slice at the 'top', 'middle', or 'bottom' of the resulting VIM ingot. The top slice was 4.25" from the top edge, the middle slice was 20.25" from the top edge, and the bottom slice was 35.25" from the top edge. Within these slices samples could either be taken from the 'middle' of the slice or the 'surface'.



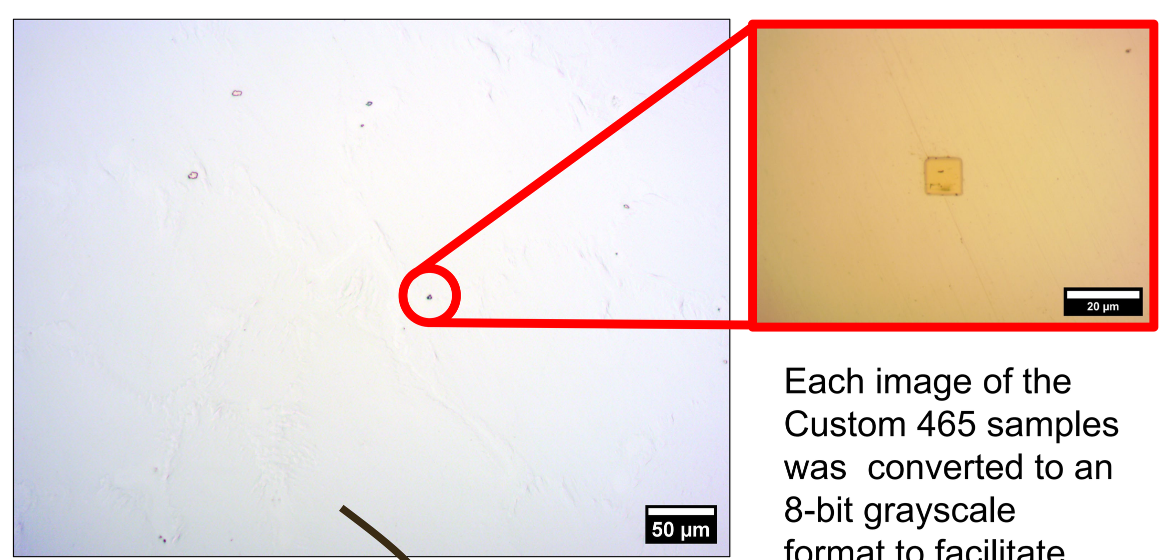
Additionally, samples would either be polished in the 'transverse' or 'longitudinal' direction of the ingot.

Individual samples were sectioned and mounted in Bakelite before being polished for optical analysis. TiN has the benefit of being visible in the matrix due to unique coloration, so etching was unnecessary. Samples were ultrasonicated in ethanol for 30 minutes for cleaning and then imaged using an optical microscope at 20x magnification. Six images were taken of each sample by moving in a 2x3 grid-like pattern to avoid unintentional bias in image selection.

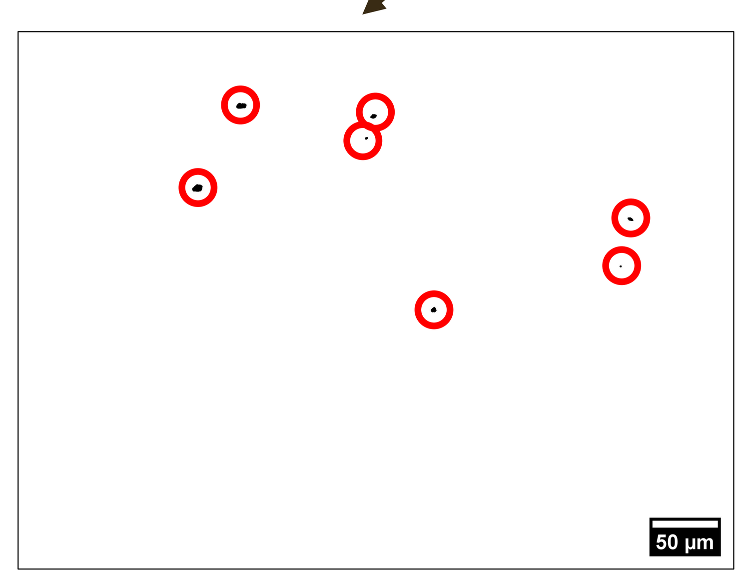




TiN Inclusion Identification



Threshold + Mask



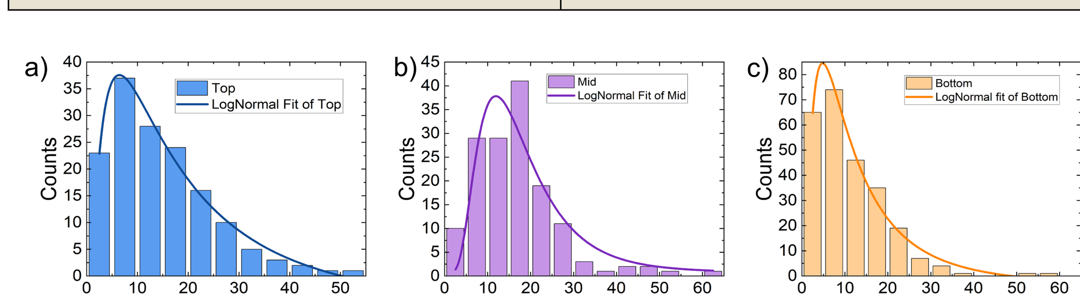
Each image of the Custom 465 samples was converted to an 8-bit grayscale format to facilitate subsequent thresholding of TiN particles. After thresholding, the images underwent manual evaluation to differentiate inclusions from optical defects. Combined with a calibration image, the size of TiN particles were measured. From this data, histograms were created and fitted to log normal distributions. Additionally, a cumulative distribution plot was created to predict the size that 97% of inclusions are smaller than.

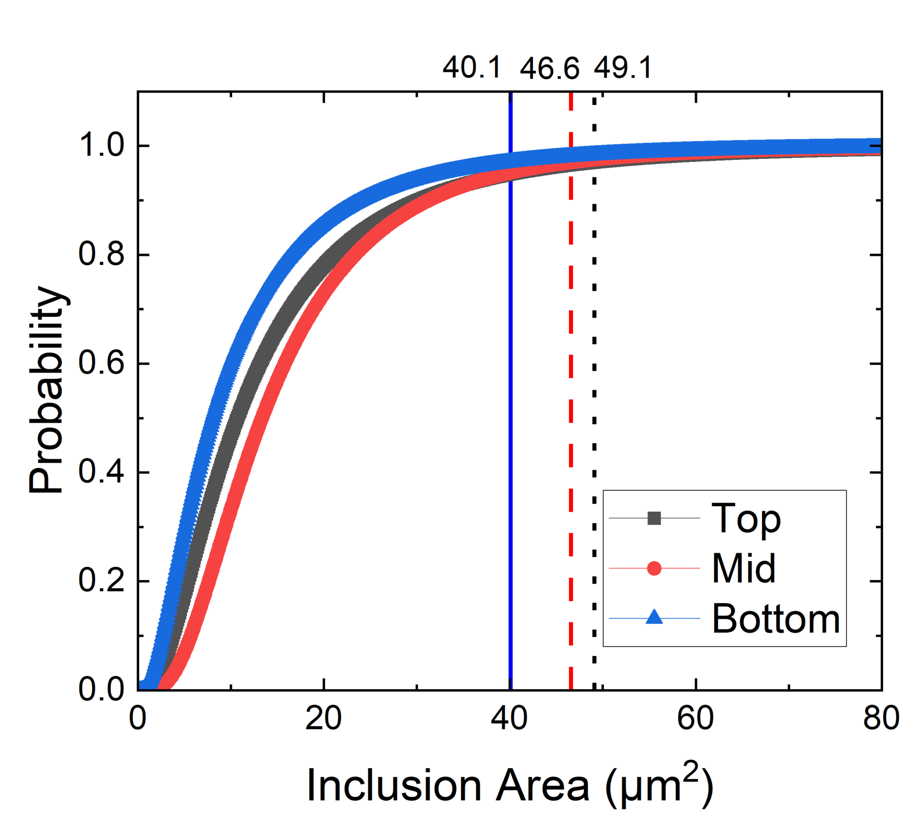
- Optical imaging provided higher image clarity compared to SEM
- TiN present with 'gold' coloration
- Clear sharp corners on cuboids

Statistical Inclusion Analysis

	Top	Mid	Bottom	Average
Inclusion Area Fraction	0.000394	0.000449	0.000413	0.000418
Average Inclusion Area (μm²)	14.12	16.19	10.98	13.76

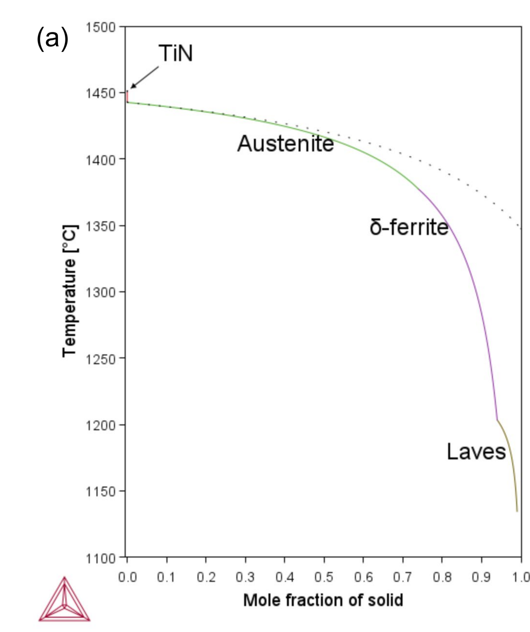
Consistent area fraction of inclusions throughout VIM ingot	Consistent inclusion size throughout VIM ingot



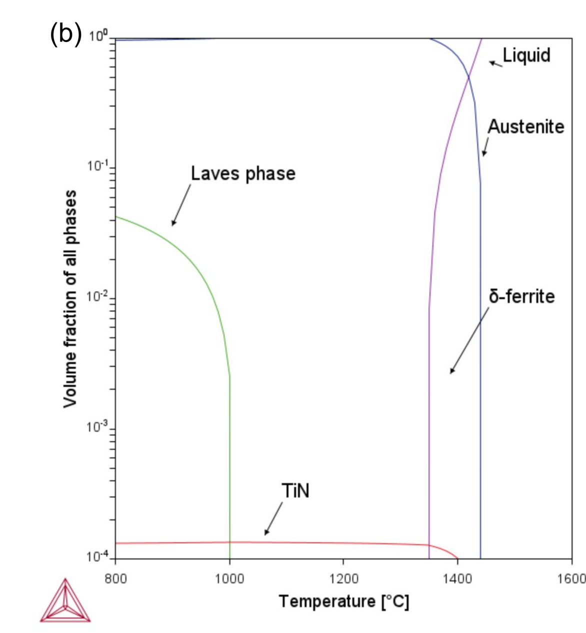


Thermodynamic Simulations

Thermo-Calc® 2023a and the TCFe8 database were used to generate a Scheil solidification diagram and an equilibrium property diagram based on composition data of Custom 465, provided by Carpenter.



Scheil solidification curve



Property diagram

Figure (a) shows the solidification sequence of the alloy. TiN inclusions form in the liquid prior to any further solidification of the alloy. As a result, it is not possible to melt, or dissolve out TiN prior to the formation of the primary microstructure of the alloy.

Figure (b) shows the volume fraction of each of the constituent phases of Custom 465, including TiN, as a function of the temperature. The volume fraction of TiN, about 0.00013, is constant until the solidus temperature, showing that it is not possible to dissolve these inclusions during conventional thermo-mechanical processing.

Conclusions

The TiN content along the length of the ingot appears to be relatively constant. The area fraction found at the top, middle, and bottom stations within the ingot are very similar, indicating that the TiN formation is independent of the longitudinal station of the ingot.

The shape of the TiN inclusions matched those found in literature, primarily showing cuboid and coalescent particle shapes. The average inclusion size is 3.71μm, assuming a cuboid shape, which falls well within the expected dimensional range of 1-5μm [2].

The thermo-calc simulation, using an equilibrium solidification solver determined that the volume fraction of TiN inclusions within the material was 0.00013, while the experimental data showed a volume fraction (average of area fraction) of approximately 0.00042.

Future Work

More analysis of inclusions on Custom 465 with iterated VAR cycles is necessary to determine how TiN inclusions vary with further processing. The methods we have established can be used to quickly assess sample surfaces and compare with the VIM only samples analyzed here. The results will show the trend of TiN formation and behavior with added VAR cycles.

The resulting trend should then be compared to the models we generated here to determine their accuracy. Using the experimental data, a more accurate model can be developed to analyze TiN behavior and ideally reduce the inclusions and their impact.

References

1. Image or Data courtesy of Carpenter Technology
2. Inada, T. (1999). *TiN inclusion formation during the solidification of stainless steel* (Doctoral dissertation, Massachusetts Institute of Technology).
3. Y. Zhu, Y.-M. Lu, C.-W. Huang, and Y.-L. Liang, "The effect of TiN inclusions on the fracture mechanism of 20CrMnTi steel with lath martensite," Materials research express, vol. 7, no. 3, pp. 036509–036509, Mar. 2020, doi: https://doi.org/10.1088/2053-1591/ab7ac3.

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