High Si concentrations in third-generation advanced high-strength steels (AHSS) are known to cause cracking in continuous cast slabs during cooling at temperatures below 300 °C. To investigate the mechanism connecting Si to this embrittlement, impact toughness tests are conducted on as-cast Fe–0.2C–3.0Mn steels with 0.5, 1.5, and 3.0 wt% Si at temperatures between 100 and 400 °C. The propagation path of the cracks through the microstructure of the specimens is examined. The ductile-to-brittle transition (DBT) behaviors of the three steels are compared. Higher Si concentrations raise the DBT temperature of the steels. The influence of Si on both solid-solution strengthening and autotempering during cooling likely contributes to this by increasing the hardness of these as-cast microstructures. In addition, the precipitation of pro-eutectoid ferrite (αPE), which is promoted by higher Si concentrations, significantly lowers the upper shelf energy of the DBT curve. The αPE phase also alters the propagation path of brittle fracture in the specimens, potentially contributing to the increase in DBT temperature. The increase in DBT temperature and decrease in upper shelf energy may contribute to the as-cast AHSS slab embrittlement at low temperatures observed in the industry.

1. Introduction

Efforts in the automotive industry to improve fuel economy and safety standards have helped spur the development of advanced high-strength steels (AHSS).\[^1\,^2\] The combination of both high strength and ductility in AHSS enables lightweighting without compromising on vehicle crashworthiness. Relatively high strength and ductility were achieved in second-generation AHSS using multiconstituent microstructures, such as the ferrite/martensite microstructure of dual-phase (DP) steels. Significant improvements in strength and ductility were made in second-generation AHSS by alloying for fully austenitic microstructures. However, the high alloy content also increased costs. Development of third-generation AHSS targets intermediate mechanical properties to the first and second generations with significantly lower alloy content and cost. The third-generation AHSS most often include high levels of retained austenite in a multiconstituent microstructure to achieve the desired properties.\[^3\]

Alloying with C, Mn, and Si, along with precise control of microstructural development during heat treatments, enables formation of third-generation AHSS microstructures. C and Mn act as austenite stabilizers, allowing the retention of metastable austenite at room temperature. Si, and sometimes Al, act as cementite growth inhibitors.\[^4\] Inhibiting carbide growth also slows tempering processes.\[^5\] The typical composition range for third-generation AHSS is 0.12–0.20 wt% C, 1.8–5.0 wt% Mn, 0.2–3.0 wt% Si, and/or 0.04–3.00 wt% Al.\[^6\,^7\]

The processing–structure–property relations for third-generation AHSS steels in their fully processed state have been widely studied.\[^11\,^12\] The exceptional properties reported for such steels have made them highly desirable for many applications, especially in the automotive industry.\[^1,^2,^11,^12\] However, processing of high-Si AHSS grades can be difficult, as the as-cast slabs often crack during cooling. There is a sizable body of literature concerning mechanisms of cracking at as-cast slabs at high temperature, above 700 °C.\[^13\,^21\] Cooling cracks are also known to occur in as-cast slabs at lower temperatures, below 300 °C, especially in higher-Si grades. The mechanism for this low-temperature cracking in high-Si as-cast slabs is not fully understood. Currently, this issue is avoided by rolling slabs without allowing them to cool below 300 °C, which can be logistically difficult and add significant cost. The current body of accessible literature on low-temperature fracture in the as-cast condition of AHSS is limited, likely due to industrial hesitancy to publish research directly related to production. Improving the understanding of the low-temperature fracture mechanisms may allow alternate, less-expensive processing routes to be found and utilized.
Si is known to influence the microstructure and mechanical properties of steels as a solid-solution strenghtener, a carbide growth inhibitor, and a ferrite stabilizer, all of which may contribute to AHSS slab embrittleduring cooling. Firstly, Si is a potent solid-solution strenghtener in steels, with 2.0 weight percent (wt%) addition increasing the yield strength of pure ferrite by 150 MPa. The reduction in dislocation mobility that often accompanies increases in strength can raise the ductile-to-brittle transition (DBT) temperature (here defined as the temperature at which brittle fracture first appears). This shift could potentially contribute to brittle crack initiation in slabs during cooling. In addition, Si is also known to greatly slow tempering in steels. Si additions may therefore result in higher strength and lower ductility in steels with a thermal processing history involving a tempering step. As-cast AHSS slabs can undergo autotempering during cooling. Therefore, the influence of Si on tempering rate may alter mechanical properties in a way that contributes to increased DBT temperature and low-temperature crack formation in AHSS slabs. Finally, Si is a ferrite stabilizer and past work has shown that it promotes the formation of proeutectoid ferrite (αPE). The αPE phase is known to occasionally initiate brittle cracks if constrained by austenite at high temperature (>700 °C) or martensite at room temperature. However, this mechanism has not been explored in as-cast AHSS microstructures (often consisting of bainite), or within the 100–300 °C temperature range the slab cracking has been observed. If the constrained αPE embrittlement mechanism is active in these systems at temperatures below 300 °C, it may be responsible for the low-temperature cracking phenomena observed. Ultimately, the contribution of each of these effects of Si to the low-temperature cracking phenomenon is not currently understood.

This work characterizes the DBT behavior of as-cast third-generation AHSS with varying Si concentrations. Literature review on the influence of Si on DBT behavior has revealed some content in the area of specialty steels for magnetic application, but the influence of Si on as-cast AHSS appears largely unstudied. Charpy-V Notch (CVN) testing, from 100 to 400 °C, was used to investigate DBT behavior. The mechanisms through which Si alters the DBT behavior are discussed, as well as how these effects could contribute to low-temperature as-cast AHSS slab cracking. CVN tests are also conducted on some tempered as-cast AHSS to explore the effects of Si on tempering in these systems.

### 2. Experimental Section

#### 2.1. Materials

Three lab-cast ingots of chemistries shown in Table 1 were used in this work. Only Si content was intentionally varied. The lab-cast compositions were within the range expected for third-generation AHSS. The ingots were induction melted in an Ar atmosphere and tapped into a cast-iron mold with a 190 by 76 mm cross section and a 264 mm height. The temperature at tapping was 1866 K (1593 °C). Each mold contained electrolytic iron chips at the bottom and was covered with a hot top after tapping. Ingots were allowed to solidify and cooled to 1070 °C within the mold before being removed and allowed to air cool to room temperature. The three ingots were produced from the same parent heat. The 0.5Si material (lowest Si) was tapped first and then Si addition was made to the melt and the 1.5Si ingot was tapped. Finally, a second Si addition was made, and the 3.0Si (highest Si) sample was tapped.

The as-cast microstructures for the three steels are shown in Figure 1. The primary microstructural constituent of all three steels was a mixture of bainitic (B) laths with interlath martensite/austenite (MA) constituent. Qualitatively, these matrix microstructures appeared to have similar lath size and phase volume fractions. The 0.5Si steel may have had a slightly finer lath size than the others, though quantitative measurements were not conducted. In addition, the 0.5Si steel had around 13 volume percent (vol%) αPE, primarily along prior austenite grain boundaries (PAGBs). The average thickness of the αPE grains was 25 μm. The 0.5Si and 1.5Si microstructures had no observable αPE. The average and standard deviation of the Vickers hardness for the 0.5Si, 1.5Si, and 3.0Si steels was 359 ± 13, 388 ± 13, and 448 ± 15 HV, respectively.

#### 2.2. Mechanical Testing

CVN tests were conducted on the 0.5Si, 1.5Si, and 3.0Si steels in the as-cast condition at 100, 200, 300, and 400 °C. The energy absorption measured from these samples, as well as inspection of the fracture surfaces and propagation paths, were used to evaluate DBT behavior. In addition, CVN specimens of the 0.5Si and 3.0Si steels were tempered at 200 and 400 °C for 30 min, cooled to room temperature, and then CVN tested at 100 °C. The tempering conditions were the same as the heat soaks used prior to the higher-temperature CVN tests. Comparison of the responses of the 0.5Si and 3.0Si steels to tempering allowed the effects of Si, which was expected to decrease the rate of tempering, to be observed. All CVN tests were conducted according to ASTM Standard E23-18 using a Tinius Olsen High Energy Pendulum Impact Testing Machine. Four specimens were tested for each composition at each testing condition. Select specimens were sectioned to reveal the fracture surface cross section. The cross sections were mounted in bakelite, ground, and polished to a 1 μm diamond finish and etched with 2 vol% nital to reveal the underlying microstructures. Fracture surfaces and microstructures were analyzed using light optical microscopy (LOM) and scanning electron microscopy (SEM). LOM images were taken with a Zeiss Axio Imager M2m upright optical microscope.

### Table 1. Chemical composition of ingots in wt% measured by optical emission spectrometry.

| Steel | C    | Mn  | Si   | Al  | P   | S   | N   | Cu  | Ni  | Cr  |
|-------|------|-----|------|-----|-----|-----|-----|-----|-----|-----|
| 0.5Si | 0.19 | 3.03 | 0.51 | 0.038 | 0.015 | 0.003 | 0.0064 | 0.021 | 0.021 | 0.05 |
| 1.5Si | 0.19 | 3.03 | 1.58 | 0.039 | 0.016 | 0.003 | 0.0063 | 0.021 | 0.021 | 0.05 |
| 3.0Si | 0.19 | 2.95 | 3.26 | 0.037 | 0.013 | 0.003 | 0.0058 | 0.020 | 0.020 | 0.05 |
SEM was performed on an FEI Quanta 200 with a field-emission gun operated at 10 kV in secondary-electron mode. Vickers hardness was measured using 10 indents made with 1 kg force and 15 s dwell time on a Zwick Materialprüfung indenter.

3. Results

3.1. As-Cast CVN Energy Absorption Results

The energy absorption results for the CVN tests on the as-cast 0.5Si, 1.5Si, and 3.0Si steels at 100–400 °C are shown in Figure 2. At 100 °C, all three steels had very low energy absorption around only 5 J. However, at 200 °C the energy absorption of the three steels was differentiated. The 0.5Si steel had the largest increase to about 50 J, the 1.5Si steel had an increase to about 21 J, and the 3.0Si steel had an increase to only 11 J. At both 300 and 400 °C, the 0.5Si and 1.5Si steels had similar energy absorptions between 57 and 68 J, indicating they had both reached the upper shelf energy before 300 °C. However, the 3.0Si steel absorption energy at 300 and 400 °C was still very low and may have still been increasing from 16 J at 300 °C to 20 J at 400 °C. From the shape of the curves, it appears that the DBT temperature for the 0.5Si and 1.5Si steels was between 200 and 300 °C, with that of 1.5Si steel being at a slightly higher temperature. The DBT temperature of the 3.0Si steel cannot be estimated from the curve alone, as no clear upper shelf energy is present.

3.2. Fracture Analysis Results

The fracture surfaces and fracture surface cross sections were examined to profile the crack propagation path and fracture mode. For all three steels at 100 °C, fracture appeared fully brittle and to follow PAGBs. At 200 °C, the 0.5Si and 1.5Si steels had a mixture of brittle and ductile fracture, with brittle fracture occurring along PAGBs in the central region of the cross section; for CVN specimens showing a mixture of brittle and ductile failure, brittle fracture is typically found in the central region of the fracture surface.[33] The 3.0Si sample at 200 °C still appeared to have majority brittle fracture along the PAGBs, possibly with a small amount of shear in the matrix directly adjacent to the crack tip. The brittle fracture appearance between the 3.0Si steel and the other two steels was significantly different, as shown in Figure 3. For the 0.5Si and 1.5Si steels, the fracture path was smooth, flat, and followed neatly along the PAGBs. The fracture path of the 3.0Si steel on the other hand was far more tortuous due to fracture along the αPE/matrix interface and cleavage fracture within...
α_{PE}, both of which were common. Additional examples of this interfacial and transgranular fracture in α_{PE} are shown in Figure 4. From this change in fracture propagation, it is clear that the presence of α_{PE} significantly altered the propagation path of brittle fracture.

Analysis of fracture mode at high temperature showed that the presence of α_{PE} significantly altered the ductile crack propagation as well. At 300 and 400 °C, the fracture in the 0.5Si and 1.5Si steels was fully ductile, microvoid coalescent, and did not follow any specific microstructural feature.
The ductile fracture regions in these steels at 200 °C also did not follow any specific feature. Fracture in the 3.0Si steel at 300 °C was found to be a mixture of brittle and ductile fracture. However, the regions of ductile fracture still followed the PAGBs and microvoid coalescence seemed to be concentrated within the $\alpha_{PE}$. At 400 °C, fracture in the 3.0Si steel appeared to be fully ductile, but continued to follow along the $\alpha_{PE}$ phase that decorated PAGBs. Figure 4 shows examples of microvoid coalescence concentrated within $\alpha_{PE}$. Based on this fracture analysis, it appears that DBT temperature of the 3.0Si steel is between 300 and 400 °C.

3.3. Tempered CVN Energy Absorption Results

The energy absorption results from the tempered 0.5Si and 3.0Si steels CVN tests conducted at 100 °C are shown in Figure 5. Tempering at 200 °C resulted in a small, but statistically significant, increase in impact toughness of the 0.5Si steel, but a negligible increase in the 3.0Si. The temper at 400 °C caused an improvement in impact toughness for both the 0.5Si and 3.0Si steels. These increases in absorption energy indicate that the material was not fully autotempered during cooling, despite the low cooling rate expected, and tempering can still affect the mechanical properties. In addition, the different effects of the tempering in the two steels demonstrate how Si delays tempering.

4. Discussion

4.1. DBT Temperature

Increasing the concentration of Si was found to increase the DBT temperature of these as-cast AHSS. The transition temperature of the 3.0Si steel was clearly highest, at between 300 and 400 °C based on the fracture surface analysis. The DBT temperatures of the 0.5Si and 1.5Si steels were both between 200 and 300 °C, though the greater ductility displayed by 0.5Si steel at 200 °C suggests that its transition occurred at a lower temperature within this range than for the 1.5Si steel.

The DBT occurs when the stress required to induce plastic deformation through dislocation glide exceeds the brittle fracture stress, as shown in Figure 6. In ferritic steels, this is typically associated with the temperature at which dislocations can no longer cross slip, thus greatly limiting plastic deformation.[4] However, other factors affecting dislocation mobility or the brittle fracture stress can influence the DBT temperature. As shown in Figure 6, increasing the yield stress of the steel can shift the DBT temperature upward, as can decreasing the brittle fracture stress.[22–26] Therefore, the increase in DBT temperature with increasing Si must be associated with the influence of Si on either the brittle fracture stress or yield stress.

Figure 5. CVN energy absorption measurements for the tempered 0.5Si and 3.0Si specimens. All tests were conducted at 100 °C. Error bars represent one standard deviation.

Figure 6. Method of defining the DBT using the intersection of the yields stress versus temperature curve and fracture stress versus temperature curve. The DBT temperature can shift to higher temperatures by increasing a) the yield stress or decreasing the b) fracture stress. Adapted with permission.[23] Copyright 2011, Elsevier.
First, the differences in DBT behavior between the as-cast 0.5Si and 1.5Si steels will be discussed, without the complicating influence of $\alpha_{PE}$. Information on the impacts of Si on yield stress is much more prevalent in the literature than any impacts on brittle fracture stress. Si is a potent solid-solution strengthen, which increases the yield stress. The tempered specimens tested in this project show that, despite the slow cooling of the ingots, the microstructures were not fully tempered. Therefore, the well-documented effects of Si on delaying tempering may also result in higher-strength matrix microstructure as AHSS slabs cool below 300 °C. The hardness of the 1.5Si steel was found to be 29 HV higher than that of the 0.5Si steel, demonstrating that the increased Si content did restrict plastic deformation. The brittle fracture within the 0.5Si and 1.5Si steels cleanly followed the PAGBs. Impurity segregation at the PAGBs is the most common explanation for this kind of failure, and no link between Si content and impurity segregation has been found in the literature. Therefore, the increase in yield stress due to higher Si levels is the most likely explanation for the increase in DBT temperature observed. Both increased solid-solution strengthening and decreased tempering of the matrix are thought to contribute, though the influence of each cannot be decoupled from the testing conducted.

The higher DBT temperature of 3.0Si steel compared with the other two steels is likely influenced both by Si effects on the properties of the matrix microstructure and by the presence of $\alpha_{PE}$. The higher concentration of Si likely resulted in an increase in the yield strength of the matrix, again due to solid-solution strengthening and a decreased degree of autotempering during cooling. However, the tortuous $\alpha_{PE}$ interfacial and transgranular brittle fracture in the 3.0Si steel, as opposed to the smooth PAGB fracture observed in the 0.5Si and 1.5Si steels, shows that the brittle fracture mechanism of the 3.0Si steel was significantly different. Therefore, the influence of increased Si on both the yield stress and brittle fracture stress on the DBT temperature must be considered.

In the literature, brittle fracture of $\alpha_{PE}$ is known to occur when the phase is encapsulated in a matrix with a much higher yield stress. The $\alpha_{PE}$ phase alone has a low yield stress. A small region of $\alpha_{PE}$ isolated within a strong matrix can have dislocation glides activated along a slip plane, yet be unable to plastically deform due to the lack of dislocation glides in the surrounding matrix material. In this situation, dislocations can pile up at the interface. The combined stress fields of the dislocations have been calculated to be high enough to locally initiate brittle fracture within ferrite. This mechanism has been documented in $\alpha_{PE}$/austenite systems at high temperatures, contributing to hot ductility troughs between 700 and 1000 °C for some steel slabs. A similar mechanism is reported at room temperature in DP steels processed to have high-strength martensitic matrixes surrounding softer ferrite. The yield strength of the bainitic matrix microstructure in these as-cast AHSS systems is much greater than that expected of $\alpha_{PE}$, especially for the highest Si concentration. Therefore, it is reasonable to assume that the brittle fracture observed within $\alpha_{PE}$ from 100 to 300 °C could be initiated by this mechanism.

However, it is unclear from these experiments if the brittle fracture stress caused by the constrained $\alpha_{PE}$ mechanism is actually lower than the PAGB without $\alpha_{PE}$. CVN tests are not particularly sensitive to the fracture stress, as they overload the specimen and only record energy absorption, which is very low for most brittle fracture. The growth of the $\alpha_{PE}$ along PAGBs could displace locally segregated impurities, resulting in the constrained $\alpha_{PE}$ mechanism causing fracture in the system, rather than impurities along PAGB, even if the two fracture mechanisms had similar brittle fracture strengths. Additional testing to determine the fracture stress of these systems with and without $\alpha_{PE}$ would be beneficial to understanding this issue.

Overall, the evidence suggests that the higher DBT temperature with increased Si content was linked to the increased yield strength of the matrix microstructure caused by Si. Both solid-solution strengthening and decreased autotempering during cooling, caused by increased Si concentration, are thought to contribute to this strengthening. The presence of $\alpha_{PE}$ may also lower the brittle fracture stress of the microstructure, shifting the DBT temperature even higher. This variation in DBT temperature may be linked to the observed failures of high-Si as-cast slabs. Though the internal stress state of slabs during cooling may be complex, it is reasonable to assume that the increased likelihood of brittle crack initiation at a higher temperature may make a slab more prone to failure.

4.2. Upper Shelf Energy

The presence of $\alpha_{PE}$ had a pronounced impact on the shape of the DBT curves. The upper shelf energy of the 0.5Si and 1.5Si steels was very similar, above 60 J, despite the differences in Si concentration. However, the upper shelf energy of the 3.0Si steel was much lower than the others, around only 20 J. The presence of $\alpha_{PE}$ in the 3.0Si, and its absence in the 0.5Si and 1.5Si steels is the most likely explanation for this. Fracture path analysis of the 3.0Si steel at high temperature shows microvoid coalescence within the $\alpha_{PE}$ across most of the fracture surface. As the crack progressed through the specimen, the $\alpha_{PE}$ and matrix ahead of the crack tip would be subjected to the stress field of the crack. The lower yield stress of the $\alpha_{PE}$ likely resulted in deformation to the point of failure within the $\alpha_{PE}$ with limited plastic deformation in the surrounding matrix, though enough to accommodate the shape change of the $\alpha_{PE}$. It is likely that the energy absorption of the 3.0Si was so low relative to the others because deformation was concentrated within the $\alpha_{PE}$, essentially limiting the volume of material available to absorb energy during the test. Strain localization within the $\alpha_{PE}$ may also be linked to the high-Si AHSS slab cracking observed in industry. Internal stresses within slabs during cooling due to thermal stresses and phase transformation may result in low and diffuse strain within slabs that lack $\alpha_{PE}$. However, when $\alpha_{PE}$ is present the strain may be concentrated within the $\alpha_{PE}$, leading to failure. In addition, strain is more likely to be concentrated within the soft $\alpha_{PE}$ phase when the matrix has a higher strength, as would be expected with higher Si concentrations.

5. Conclusion

1) Increasing Si concentration was found to increase the DBT temperature of as-cast AHSS. Increasing Si resulted in increased...
hardness of the matrix microstructure, likely due to both solid-solution strengthening and the reduction in the degree of autotempering induced by Si. The increased yield strength of the microstructure was a strong contributing factor to the increase in DBT temperature; 2) The constrained ferrite embrittlement mechanism, typically associated with slab cracking at high temperature (hot ductility trough), was found to be the primary cause of brittle fracture at temperatures below 300 °C in the 3.0Si (highest Si) as-cast AHSS. Activation of this embrittlement mechanism may have also contributed to increased DBT temperature in the highest Si as-cast AHSS composition; 3) The αPE phase, promoted by higher Si levels, resulted in a significant decrease in the upper shelf energy of the DBT curve. The low yield stress of the αPE relative to the matrix was found to result in strain localization within the αPE, decreasing the volume of material undergoing plastic deformation and therefore the overall energy absorption of the steel; and 4) Increased Si levels can delay the rate of tempering in these as-cast AHSS at low temperatures (200 °C). This indicates that Si can slow the rate of autotempering in AHSS slabs during initial cooling, possibly contributing to lower ductility and crack initiation in industry.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords
as-cast mechanical properties, ductile-to-brittle transitions, proeutectoid ferrite, silicon, slab cracking, third-generation advanced high-strength steels

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