

## Thermal Processing and Alloys Selection to Modify Steel Fiber Performance in Ultra-High Performance Concrete

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### Abstract

This research aims to characterize the microstructure and the mechanical behavior for a variety of alloys including identification of heat treatments to modify stress vs. strain behavior. Four different fibers were studied with varying shapes and alloy compositions. Fibers were tensile tested to determine the mechanical properties. Fractography and microstructures were characterized using scanning electron microscopy (SEM). The fibers were heat treated by three different methods: fully annealed, partially austenitized, and fully austenitized material. Heat treatments were conducted to manipulate the mechanical and microstructural composition and investigate how they affect positively or negatively their properties. The mechanical performance and microstructure of the fibers were analyzed in both the as-received material and after being heat treated. Fibers were compared by their mechanical performance and viability of heat treatment processes.

Key Words: Ultra-high performance concrete, fiber reinforcement, carbon steel, stainless steel, heat treatment, tensile tests, microstructure

### 1. Introduction

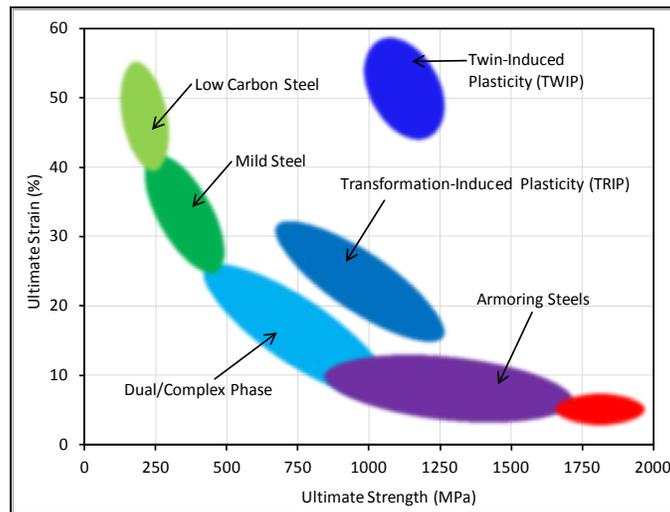
Metallic fiber reinforcement is used ubiquitously in cementitious composite materials such as UHPC at volume fractions of 1-3% of the total mixture proportion. Fibers range in length from 10mm-40mm with various morphology including straight, hooked, and helical shapes. The diameter of fiber reinforcement typical ranges from 0.2mm-0.5mm [1]. One of the inherent limitations of typical metallic reinforcement results from the way it is produced. The heavy cold drawing required to produce small diameter fibers results in extremely high tensile strengths (on the order of 2,000MPa) and low ductility of 1-5%; essentially a brittle material. To impart improved toughness in composite materials, increased ductility of fiber reinforcement is necessary. While this may be achieved by heat treating (e.g., annealing) fibers following cold drawing, this would sacrifice tensile strength. Innovative materials are required that provide additional plastic deformation mechanisms beyond traditional dislocation nucleation and motion to offer both high strength (1,000-2,000MPa) and high ductility (25-75%).

Recent research and development efforts by metallic alloy producers have led to the development of a variety of high-performance metallic materials that exhibit the combined properties of high strength and toughness [2]. The current focus of this R&D is on components for the automotive industry such as improving the energy dissipation of vehicle bumpers. These materials take advantage of complex stress-activated phase transformation deformation mechanisms such as the transformation from face-centered cubic austenite ( $\gamma$ ) to body-centered

tetragonal martensite ( $\alpha'$ ), resulting in a volumetric expansion. By controlling chemical composition, alloys can be produced that exhibit stacking fault energies (SFE) indicative of metastable microstructures (SFE less than 20mJ/m<sup>2</sup>) that can readily transform upon the application of stress [3]. Similar modifications in chemical composition can also impart the potential for twin generation with the application of stress [4]. Previous research has shown that these metallic materials that exhibit stress-activated phase transformations can provide up to a 100% increase in ductility while maintaining high tensile strengths (up to 1,500MPa) when compared with typical metallic alloys used for fiber reinforcement [5] (see TRIP and TWIP regions in Figure 1).

Typical plastic deformation mechanisms in metallic alloys are kinetic in nature with physical motion of dislocations occurring. Because of this, these materials are sensitive to strain rate and generally exhibit moderate increase in tensile strength and reductions in ductility as strain rate increases to dynamic levels (i.e., in excess of 10<sup>2</sup> s<sup>-1</sup>). Since phase transformation and twin-induced deformation mechanisms are nearly diffusionless and only require minor modifications to crystallographic lattice structures, their mechanical response is not affected by strain rate, even at strain rates up to 10<sup>4</sup> s<sup>-1</sup> [6] (see Figure 2). This unique behavior at high strain rates portends to the applications for facility protection where extreme load cases may include high strain rate impact and penetration events.

While the initial R&D and basic research on stress-activated phase transformation and twinning materials has provided a substantive starting point for the proposed research, it has largely focused on metallic components for the automotive industry [7]. There exists a limited understanding of their behavior at blast and impact relevant strain rates, complex stress states, and under cyclic loading [8]. The research described herein addresses this limited knowledge by studying a variety of fiber shapes and alloys compositions. In addition, we have identified heat treatments which can be used to modify the stress vs. strain behavior of steel fiber reinforcement in UHPC. Microstructural characterization studies were performed along with mechanical testing.



**Figure 1. Comparison of ultimate strain vs. ultimate strength for various families of metallic materials (from [9]).**

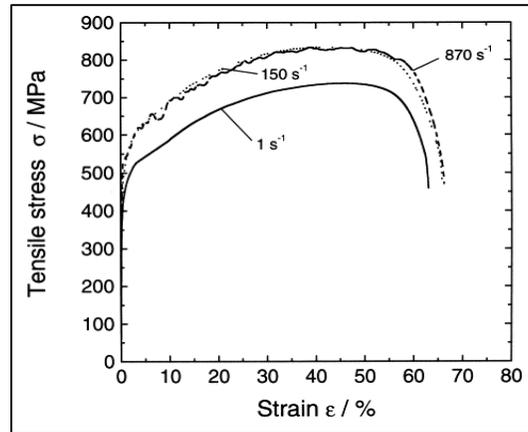


Figure 2. Influence of strain rate on stress vs. strain behavior of TWIP steel (from [6]).

## 2. Methodology

This research is focused on analyzing thermal processing parameters and their influence on mechanical behavior and fracture mechanisms. The materials analyzed are a diverse set of existing fibers produced from various alloys (stainless steel and carbon steel) for reinforcement of cementitious composites. The existing fibers studied have different shapes: kinked and undulated as shown in Figure 3. It is necessary to quantify the mechanical properties and characterize the fracture mechanisms of each material such that a suggested material with a specific heat treatment that can achieve a high energy dissipation with a greater ductility can be identified.

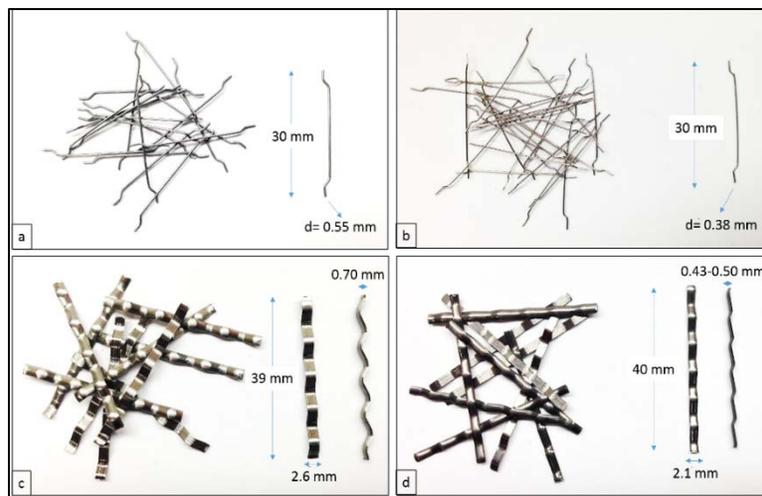


Figure 3. Existing fibers dimensions: a) kinked carbon steel, b) kinked stainless steel, c) undulated stainless steel, d) undulated carbon steel.

### 2.1 Energy Dispersive Spectroscopy (EDX)

Since manufacturers do not specify the material of what the fibers are made, an EDX analysis is performed in order to give a rough material composition. For the stainless steel fibers the EDX was used to quantify the weight percent of nickel (Ni) in the material as well as other elements including Fe, Cr, Mo, and Mn. Ni and Mn are key elements that favor the FCC structure of austenite and control austenitizing ability of the stainless steel by annealing.

## 2.2 Annealing

Conventional kinked fibers were heat treated in order to modify the mechanical properties. The kinked carbon steel fibers were annealed at 800°C and the stainless steel fibers were annealed at 1000°C. Both of them for 6 minutes holding time and 60 min holding time. Further tensile tests were performed in order to compare them with the as received materials.

## 2.3 Tensile Testing

A tensile test was conducted using an Instron E300 at a quasi-static strain rate ( $0.00001 \text{ s}^{-1}$ ) in order to measure the ductility and strength of each material. Figure 4 presents the samples after being tested and prepared on a sample holder for scanning electron microscopy imaging (i.e., fractography). The samples were tested as received and after the heat treatment. This will be used to compare the ductility and strength transformation.



**Figure 4. Fibers after tensile test.**

## 2.4 Fractography using Scanning Electron Microscopy (SEM)

An FEI Nova NanoSEM 630 will be used to characterize the fracture surface on the tensile tested sample. Fractography analysis will be used to determine the failure mode and the influence of material processing and alloying on fracture.

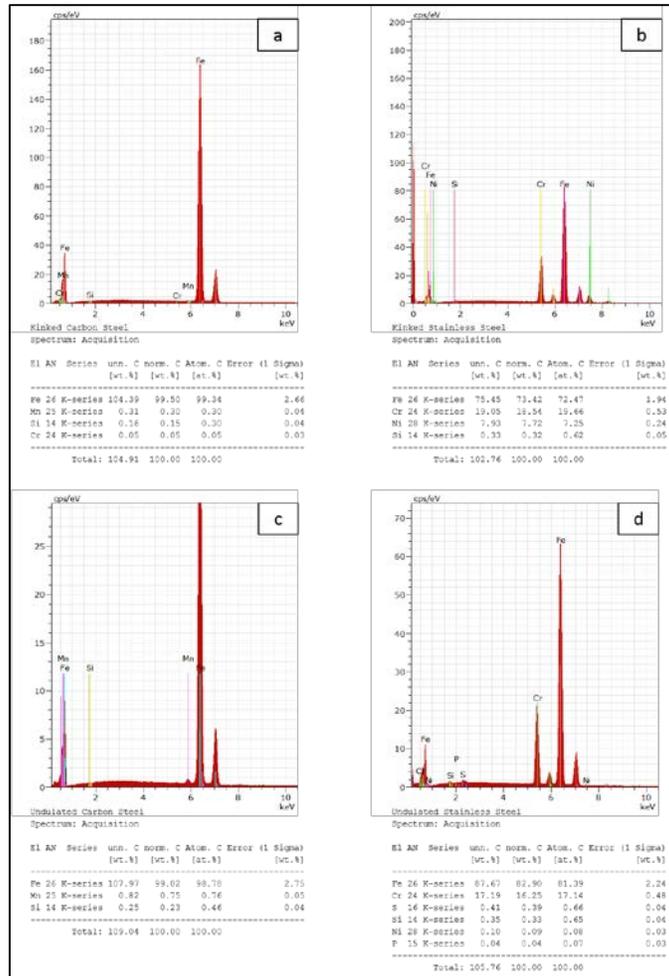
## 3. Preliminary result and discussion

### 3.1 Energy Dispersive Spectroscopy (EDX)

The EDX gave a rough composition of the materials as shown Figure 5. Analyzing the chemical composition data and the tensile tests performed to as receive material it was able to presume that the kinked stainless steel fibers are AISI301. The chemical composition collected by the EDX of the kinked stainless steel presented in Figure 5b matched with chemical composition of AISI301 presented in Table 1. Otherwise, the EDX analysis for the undulated stainless steel fiber (Figure 5d) do not corroborate with the AISI301 chemical composition and also do not have enough Ni to support the presence of austenite. For this reason and the further presented tensile data it was decided to focus the study in the kinked fibers and omit the undulated fibers.

**Table 1. Chemical composition of 301 SS (Balance of Fe)**

Carbon	Chromium	Nickel	Silicon	Manganese	Molybdenum
Max 0.12 wt%	16-18 wt%	6.5-0 wt%	<1.5 wt%	<2 wt%	<0.8 wt%



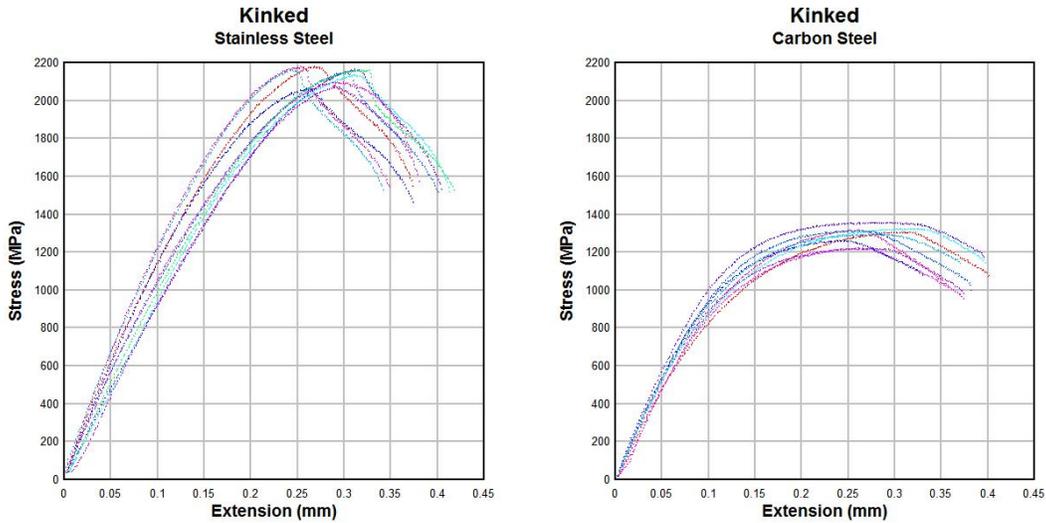
**Figure 5. EDX results for: a) kinked carbon steel, b) kinked stainless steel, c) undulated carbon steel, d) undulated stainless steel.**

### 3.2 Tensile Test Including Heat Treatment Effects

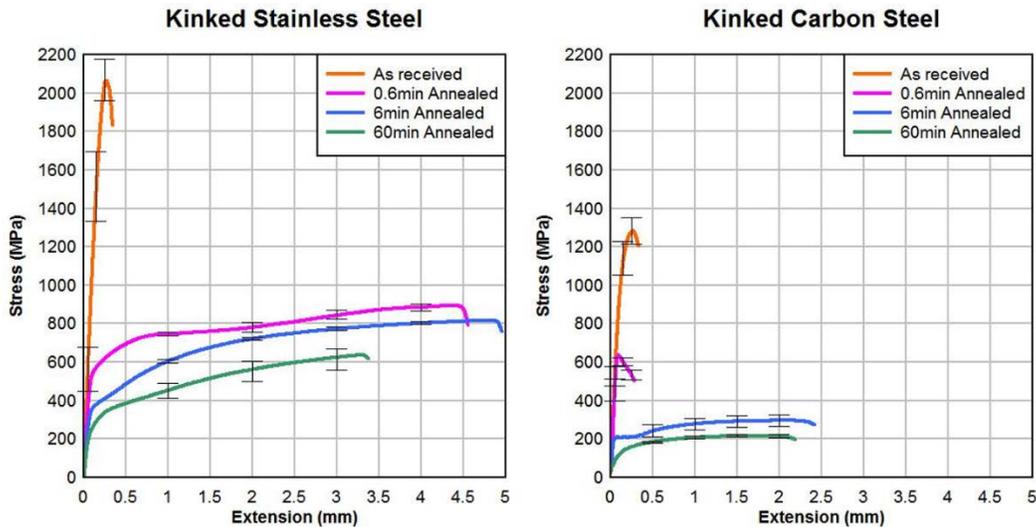
The samples tested presented different mechanical behavior depending on the alloy and the fiber morphology (i.e., size and shape). Refer to Figure 6 for plots. The as received kinked fibers indicated that stainless steel is stronger than the carbon steel fiber. Stainless steel having an average of ~2150 MPa, compared to carbon steel with an average of ~1280 MPa. Both materials are heavily cold drawn into wire, resulting in the observed strengthening over a fully-annealed condition. The ductility of the kinked fibers of both alloys was similar, ~0.25mm of extension. Evidence of strain localization immediately following yielding was observed in the stainless steel fibers. This localization was not present in the carbon steel fibers which due exhibit some global plasticity prior to localization and failure. For the undulated samples, it is difficult to compare mechanical behavior between the different alloys that were tested because for a same type of fiber (carbon steel or stainless steel) the thickness and wide dimensions varied greatly.

After the fibers were annealed for different time durations, mechanical testing was performed with the intent to quantify the mechanical behavior of the heat treated materials. Refer to Figure 7 for a comparison of mechanical behavior for the heat treatment regimes studied. For both materials, 6 min annealed present a higher extension. The shortest annealing time used did

not fully austenitize the structure. Longer annealing times likely resulted in grain coarsening, resulting in a loss of strength and ductility. For the stainless steel, comparing the as received with the 6 min annealed fiber, the extension was ~0.25 mm and ~4.6 mm respectively, resulting in the 6 min annealed fibers being 18.4 times more ductile. The carbon steel fiber with 6 min of annealing increases 8.9 times in ductility over the as-received fiber.



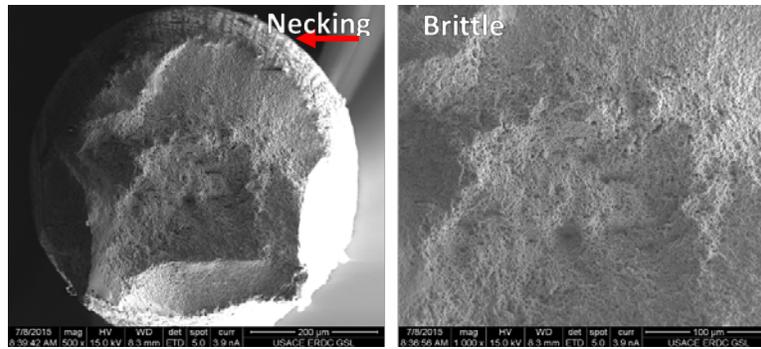
**Figure 6. Kinked fiber comparison.**



**Figure 7. Mechanical comparison of fiber including heat treatment.**

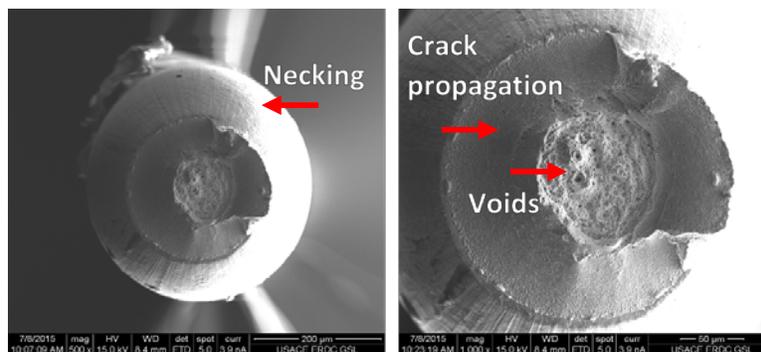
### 3.3 Fractography

After the fibers were tested in tension, fractography was performed using SEM. The carbon steel fiber, shown in Figure 8, presented a brittle fracture with a small central ductile surface. This is a characteristic of carbon steel materials that are highly cold drawn. The thin area around the sample where the necking starts is shown.

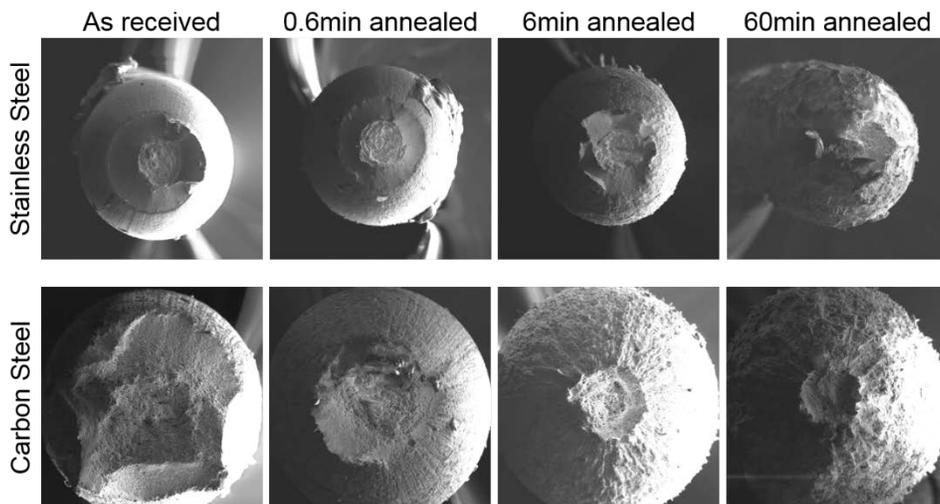


**Figure 8. Kinked carbon steel fiber fractography.**

The stainless steel fiber had a ductile fracture mode with nucleation of voids followed by their growth and then coalescence. In Figure 9, a larger necked area is shown along with vast voids in the center of the sample that cause the fracture propagation. The sample reduced 63% of the diameter before failure, compared with carbon steel that reduced only 26% of its diameter. The asymmetric crack can be attributed to a pore or a non-uniform surface. Fractography was also performed for the annealed kinked fibers following mechanical testing. As can be observed in Figure 10, ductile failure dominates the fracture surface as annealing time increase. It is evident the increase in the necking area as increasing annealing time.



**Figure 9. Kinked stainless steel fiber fractography.**



**Figure 10. Fracture surface as annealed increment.**

#### 4. Conclusion

Stainless steel fibers which were more heavily cold drawn exhibited higher strength than carbon steel fibers but limited plasticity which localized quickly. Undulated fibers behavior much different than the kinked steel fibers that include a straight gage section. Variability was due to the irregular shape of the undulated fibers as well as inconsistency in the fiber size. Chemical analysis of the fibers was used to identify heat treatability as well as appropriateness of the chemical composition for inducing TRIP behavior. The chemical composition of the undulated stainless steel fiber did not include a high enough content of austenite stabilizer Ni, limiting its viability for inducing TRIP deformation mechanisms. Heat treatment had a tremendous influence of stress vs. strain behavior with reductions in yield and ultimate strength along with approximately an order of magnitude increase in ductility. More importantly, strain hardening was introduced to the behavior, which has a potential to modify fiber pullout behavior and impart new energy dissipation mechanisms in composite materials. A 6 minute annealing time was identified to be optimal for the alloys studied. This resulted in a decrease in strength and increase in ductility with limited coarsening of grains. The stainless steel fibers exhibited the greatest strain hardening ductility, likely due to the combination of conventional deformation mechanisms (e.g., dislocation nucleation and motion) along with TRIP mechanisms. Fractographic analysis confirmed the trends observed in mechanical behavior differences based on alloy composition and increasing plasticity due to heat treatment.

#### 5. Acknowledgements

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