

# Effects of Matrix Microstructure on the Nanoscale Precipitation and Precipitation Strengthening in an Ultra-high Strength Steel

Songsong Xu, Hao Guo, Yu Zhao, Naimeng Liu, Dan Chen, Ye Cui, Yang Zhang and Zhongwu Zhang

**Abstract** Matrix microstructure and nanoscale clusters are the two main factors influencing the mechanical properties of nanocluster strengthened steels. Here, an ultra-high strength steel with a tensile strength of  $\sim 1.64$  GPa and an elongation of  $\sim 14\%$  has been developed through a combination of fine matrix microstructure and precipitation strengthening. Matrix microstructure was primarily controlled by annealing treatment. After annealing treatment at  $750\text{ }^{\circ}\text{C}$  for 1 h, the hot-rolled microstructure changes to the layered sorbite-like structure. The precipitation strengthening contributes a similar yield strength of  $\sim 494$  MPa in both hot-rolled and annealed steels. The results indicate that there is no effect of matrix microstructure on the subsequent precipitation of nanoscale clusters and precipitation strengthening. The matrix microstructure and the precipitation of nanoscale clusters are independent and can be controlled separately.

**Keywords** Ultra-high strength steel · Matrix microstructure · Precipitation strengthening

## Introduction

There has been an increasing demand of ultra-high strength steels (USS) with characteristics like high strength, good weldability and corrosion resistance due to the rapid economic development and fast growing national defense industry [1–4]. Precipitation strengthening plays an important role in the design and fabrication of USS with superior mechanical properties and strong resistance to radiation damage [5–12]. To obtain a good comprehensive properties, Cu and NiAl nanoparticles are two classes of effective strengthening phases among the various types of potential

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nanoparticles to be considered for precipitation hardening in steel [13, 14]. Jiao et al. [15] has found that Fe–Cu–Ni–Al-based steels with relatively low Ni/Cu and Al/Cu ratios, in which Cu nanoparticles are the dominant strengthening phase and NiAl nanoparticles provide a supplement strengthening effect. And if small variations in the Ni, Al and Cu contents, precipitation characteristics would have sensitive differences, including the composition, size and number density of the Cu and NiAl nanoparticles, thereby significantly influencing the degree of strengthening.

Although Cu-riched nanoscale precipitation appeared in the USS, can increase the mechanical properties and solve the problem of weldability [14, 16–19], the demand for the matrix microstructure in it is accordingly strict. Furthermore, the USS' matrix microstructure will be changed with the annealing treatment, variation of subsequent cooling rates, and alloy chemistry [14]. By austenite decomposing during continuous cooling rate, Thompson [20] has made the CCT diagram for the HSLA-80 steel and found that the austenite would transform to polygonal ferrite, widmanstatten ferrite, granular ferrite, acicular ferrite, upper bainite, lower bainite and martensite. By varying the continuous cooling rates and using three-dimensional electron backscatter diffraction and transmission electron microscopy, Kang [21] investigated that granular bainite, acicular ferrite and lath-type bainite will appear in the HSLA steel at the different cooling rates. However, understanding the relationship between annealing treatment and the precipitation of nanoscale clusters is the most important prerequisite for the preparation of nanocluster-strengthened steels with a combination of high strength and ductility.

In this paper, an experimental study is reported on for the precipitation strengthening in an ultra-high strength steel with different matrix microstructure. Combining the mechanical properties and matrix microstructure, the effect of matrix microstructure on the precipitation of nanoscale cluster was conducted.

## Experimental Methods

A nanoprecipitates strengthened steel with a nominal composition of Fe-1.5Mn-2.5Cu-4Ni-1Al-0.005B-1.5Mo-0.05Nb-0.1Ti-1.5W-0.08C-0.5Si (wt%) was selected for the experimental investigation. The cast ingots with a weight of ~50 kg were obtained by vacuum induction melting with magnetic stirring to ensure homogeneous distribution of alloying elements. The cast ingots were rolled to a total reduction of 80% with a temperature of 800 °C through 8 passes. The final thickness of the as-rolled steel plate was 12 mm. The hot-rolled sample is labeled as HR. To identify the effects of annealing treatment on the precipitation of nanoscale clusters, HR steel was then annealing at 750 °C for 1 h after removing the surface oxides (labeled as 750AS). And then all the samples, HR and 750AS were aged at 525 °C for 2 h. The aged samples are labeled as HR-AG and 750AS-AG, respectively.

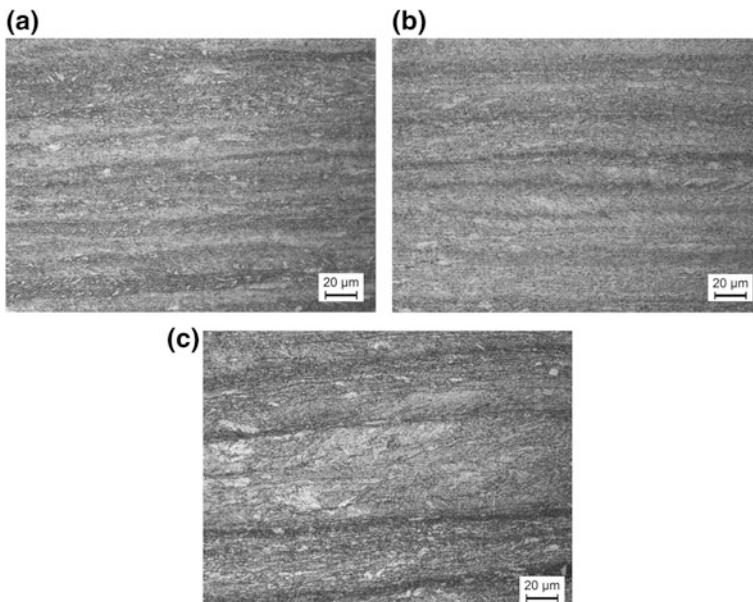
Hardness measurements were conducted on a Vickers tester with a load of 1 kg for 15 s. For each specimen, at least ten indents were measured to obtain an average value. The matrix microstructures were characterized using optical microscopy

(OM). The OM samples were polished to a final surface finish of  $0.2\ \mu\text{m}$  by standard mechanical polishing procedures, and then etched for approximate 5–15 s with a 4 vol.% Nital solution.

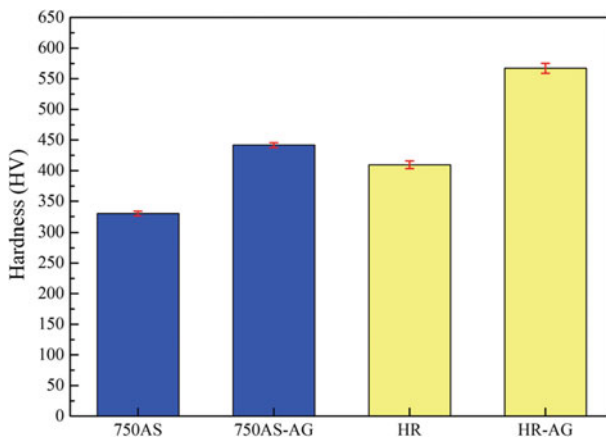
Tensile tests along the rolling direction of the samples were conducted on an Instron 5565 testing machine at a strain rate of  $10^{-3}\ \text{s}^{-1}$ . The rod tensile samples were processed into the specimens with 5 mm in diameter and 25.4 mm in gauge length by numerically controlled lathe. Three specimens were tested in the same condition and the average values were reported. A contacting Instron extensometer was used to measure strain within the sample gauge upon loading. The yield strength was determined with the 0.2% offset plastic strain method.

## Results and Discussion

Figure 1 shows the optical microstructures of HR and 750AS steels. The HR steel consists of the deformed original austenite and some recrystallized ferrites embedded in the deformed matrix (Fig. 1a), indicating that dynamic recovery/recrystallization is the predominant softening mechanism. After annealing at  $750\ ^\circ\text{C}$  for 1 h followed water quenching, the deformed fibrous structure of HR steel transformed into the layered sorbite due to recrystallization (in the Fig. 1b). And the layered sorbite were introduced long the rolling direction, the groups of sorbite were thin and homogeneous. The orientation of sorbite showed a symmetry



**Fig. 1** The optical microstructures of **a** HR steel, **b** 750AS steel, **c** 750AS-AG steel

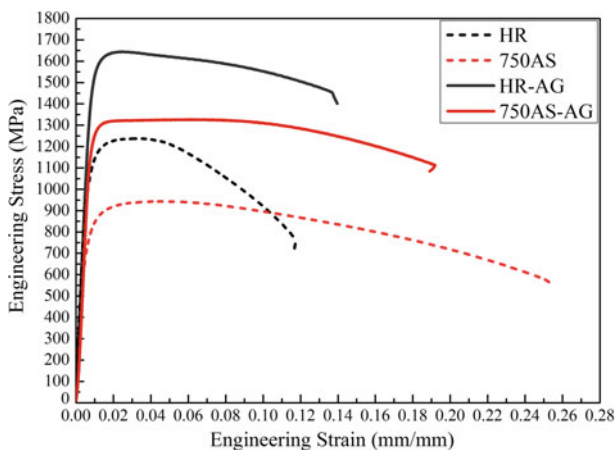


**Fig. 2** Microhardness of HR and 750AS steels before and after aging treatment

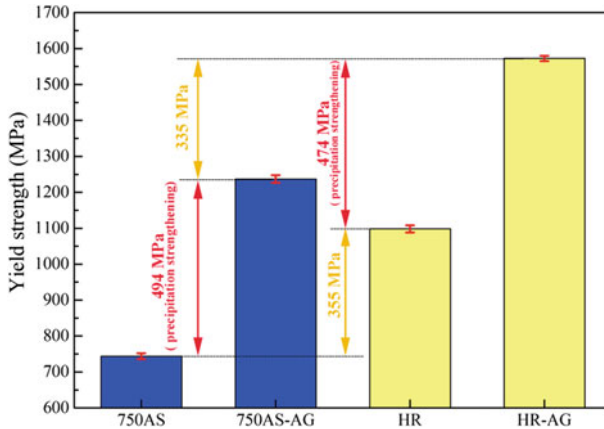
along the boundary between the layers. With aging at 525 °C for 2 h, the 750AS-AG steel was also consisted of sorbite in the Fig. 1c. It confirms that aging treatment has no effect on the microstructure.

Microhardness measurements were conducted to evaluate the age hardening response of the HR and 750AS steels. The microhardness values were shown in Fig. 2. After aged at 525 °C for 2 h, the microhardness of HR and 750AS steels all has obviously increase due to the formation of nanoscale precipitates. The hardness increased significantly from 409 to 567 HV at an aging time of 2 h for HR steel, while 750AS steel has an increase of 111 HV. However, the hardness of 750AS steel has an obvious reduction than the HR steel due to the recrystallization. Aging treatment has no influence on the above results.

The engineering stress-strain curves were presented in Fig. 3. The HR steel showed a yield strength of ~1098 MPa and an ultimate tensile strength of



**Fig. 3** Room-temperature tensile stress-strain curves of the steels



**Fig. 4** Schematic diagram showing the effects of precipitation strengthening

~1237 MPa. As for HR-AG steel, the yield strength increased dramatically to ~1572 MPa with an elongation-to-failure 14%. Owing to the change of microstructure, the yield strength of 750SS steel has an obvious reduce from 1098 to 743 MPa comparing with HR steel. As for 750SS-AG steel, it has an increase of yield strength from 743 to 1237 MPa. As shown in Fig. 4, for all the two samples, HR and 750SS steels, the increments of yield strength induced by aging were very similar (474 MPa for HR steel, 494 MPa for 750SS steel). Owing to the different microstructure induced by different processing routes, matrix microstructure contributes different yield strength to HR and 750AS steels. However, the contributions of precipitation strengthening were similar for HR-AG and 750AS-AG steels. These results indicated that matrix microstructure had no effect on precipitation of the clusters and precipitation strengthening in the ultra-high strength steel.

As compared to the HR sample, the yield strength of 750AS decreased 355 MPa due to the change of matrix microstructure. However, the yield strength increment of HR and 750AS after aging were comparable though two sample have different matrix microstructure. These indicated that there was no effect of matrix microstructure on the precipitation of nanoscale clusters and precipitation strengthening, and the precipitation of nanoscale clusters and annealing treatment were independent. Our previous studies also confirmed this by in similar steels [9]. Local composition fluctuation would form Cu-enriched embryos and Cu-depleted regions, which are not rely on the matrix microstructure which was effected by the annealing treatment. The embryos become preferential sites for nucleation of nanoscale clusters when they reach a critical size.

## Conclusion

In summary, the ultra-high strength steel with a tensile strength of 1.64 GPa and an elongation of  $\sim 14\%$  have been successfully developed through a combination of fine matrix microstructure and nanoscale clusters. The matrix microstructure was controlled by the annealing treatment, which has no effect on the precipitation of nanoscale clusters. The matrix microstructure and the precipitation of nanoscale clusters are independent and can be controlled separately.

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