Effects of welding and post-weld heat treatments on nanoscale precipitation and mechanical properties of an ultra-high strength steel hardened by NiAl and Cu nanoparticles

Z. B. Jiao¹, J. H. Luan¹, W. Guo², J. D. Poplawsky², C. T. Liu^{1,*}

¹ Center for Advanced Structural Materials, Department of Mechanical and Biomedical Engineering, College of Science and Engineering, City University of Hong Kong, Hong Kong, China

² Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

Abstract

The effects of welding and post-weld heat treatment (PWHT) on nanoscale co-precipitation, grain structure, and mechanical properties of an ultra-high strength steel were studied through a combination of atom probe tomography (APT) and mechanical tests. Our results indicate that the welding process dissolves all pre-existing nanoparticles and causes grain coarsening in the fusion zone, resulting in a soft and ductile weld without any cracks in the as-welded condition. A 550 °C PWHT induces fine-scale re-precipitation of NiAl and Cu co-precipitates with high number densities and ultra-fine sizes, leading to a large recovery of strength but a loss of ductility with intergranular failure, whereas a 600 °C PWHT gives rise to coarse-scale re-precipitation of nanoparticles together with the formation of a small amount of reverted austenite, resulting in greater recovery in both strength and ductility. Our analysis indicates that the degree of strength recovery is dependent mainly upon the re-precipitation

* Corresponding author: E-mail: chainliu@cityu.edu.hk, Tel: 852-34427213

microstructure of nanoparticles, together with grain size and reversion of austenite, while the ductility recovery is sensitive to the grain-boundary structure. APT reveals that the grain-boundary segregation of Mn and P may be the main reason for the 550 °C embrittlement, and the enhanced ductility at 600 °C is ascribed to a possible reduction of the segregation and reversion of austenite.

Keywords: welding; ultra-high strength steel; precipitation; mechanical property; structure-property relationship

1. Introduction

Ultra-high strength steels have received a growing interest in recent years due to their high potential for weight reduction and performance improvement for various lightweight applications, such as aerospace, vehicles, shipbuilding, and wind energy. In the fabrication of these structures, welding is one of the most important and versatile processes available to industry for joining steels, and a good weldability is a highly desirable feature of ultra-high strength steels. From a metallurgical point of view, although carbon is one of the most potent strengthening elements in steels, too much carbon is extremely harmful to weldability, through increasing the probabilities of forming brittle martensite and hence the risk of cold-cracking after welding [1]. As such, ferrous metallurgy emphases have been increasingly placed on the development of ultra-high strength steels with reduced carbon contents for improving weldability. Nanoparticles strengthened ferritic/martensitic steels represent an important class of advanced steels which, by virtue of appropriate composition and thermo-mechanical processing, exhibit both high strength and good ductility while minimizing the use of carbon and other alloying additions [2-8]. In particular, the precipitation of coherent nanoparticles, such as body-

centered cubic (bcc) Cu-rich nanoclusters [2-5,9-12] and B2-ordered NiAl intermetallic nanoparticles [13-17], is very appealing, because they can precipitate on a sufficiently fine scale (less than 5 nm in diameter), providing an extremely high strengthening response. Currently, research is starting to emerge on the weldability of this class of high-strength steels, mostly of Cu-rich nanoclusters strengthened steels [18-23]. Previous welding studies showed that the hardness of heat-affected zone (HAZ) and fusion zone (FZ) of Cu-strengthened steels was under-matched to the base metal (BM), which was considered to be due to the partial dissolution of pre-existing Cu-rich nanoclusters within the HAZ and full dissolution of nanoclusters within the FZ, as characterized by atom probe tomography (APT) [18-21]. It has been found that the re-precipitation of nanoparticles can be achieved by designing an appropriate post-weld heat treatment (PWHT) or multi-pass welding procedure, which provides a promising way to recover the mechanical properties of welds [22,23].

Recent studies reveal that the co-precipitation of multiple types of nanoparticles is more attractive, as compared with precipitation of a dispersion of a single type of nanoparticles, since the co-precipitation approach may lead to a superior combination of different properties resulting from the synergistic combination of multiple types of nanoparticles with different compositions, microstructures, and micromechanical properties [24-30]. For example, through the optimization of alloy compositions, the co-precipitation of Cu-rich and NiAl-based nanoparticles can be achieved, which offers a promising way to effectively strengthen steels to as high as 2000 MPa while maintaining a good ductility of approximately 10% elongation [28,29]. To date, however, the welding properties of this new class of nanoscale co-precipitation strengthened steels is poorly understood, and their post-weld microstructure-property relationship remains unclear. For an understanding of and eventually control over the microstructure and mechanical property

response to welding, it is of fundamental importance to understand the effect of welding and PWHT on the nanoscale co-precipitation, grain structures, and mechanical properties of this new class of nanoscale co-precipitation strengthened steels with ultra-high strength.

The purpose of this study is to elucidate the mechanism of the precipitate microstructure and grain structure evolution during welding and PWHT, and further to correlate the microstructure with bulk mechanical properties of a NiAl/Cu co-precipitation strengthened steel. The precipitates characteristics, including the size, number density, composition, and solute partitioning behavior of NiAl and Cu co-precipitates were thoroughly investigated by APT. Particular attention was paid to understanding the basic mechanism involved in the precipitate dissolution and re-precipitation and to further elucidate the key factors governing the weld strength and ductility.

2. Experimental

The material investigated in this study is a high-strength steel with a composition of Fe-5Ni-2Al-3Mn-1.5Cu-1.5Mo-1.5W-0.07Nb-0.05C-0.01B (wt.%) [29]. Alloy ingots were prepared by arc-melting a mixture of the constituent elements with purity better than 99.9 wt.% in a Ti-gettered high-purity argon atmosphere. Repeated melting was carried out at least five times to ensure the chemical homogeneity. The melted alloys were then drop-cast into a copper mold with dimensions of $15 \times 30 \times 100 \text{ mm}^3$. The resulting plates were cold rolled by multiple passes with a total reduction of ~66% and then solution-treated for 30 min at 900 °C, followed by water quenching, and then aged isothermally at 550 °C for 2 h, resulting in the formation of ~3-nm NiAl and Cu co-precipitates [29].

4-mm-thick plates were welded using the gas tungsten arc welding (GTAW) technique, in which an arc was established between a non-consumable tungsten electrode and the work piece in an ultra-high purity argon atmosphere. The welding was performed with a welding power of ~ 2 kW and a travel speed of ~ 3 mm/s. The as-welded plates (which will be called hereafter the AW state) were post-weld heat treated at 550 °C and 600 °C for 30 min, which will be referred to as PWHT-550 and PWHT-600, respectively.

Transverse cross-sections of the AW, PWHT-550, and PWHT-600 samples perpendicular to the welding direction were polished to a final surface finish of 0.05 μm using standard mechanical polishing procedures. Hardness measurements were conducted on the polished surface of the samples using a Vickers hardness tester with a load of 2 N for 15 s. Tensile samples with a cross-section of $3.2 \times 1 \text{ mm}^2$ and a gauge length of 15 mm were cut by electro-discharge machining, and ground careful on each side with SiC paper through 4000 grit. Tensile tests along the rolling direction of the samples were conducted on an MTS tensile testing machine at a strain rate of 10^{-3} s^{-1} . Two specimens were tested for each condition, and the average values were reported. Fracture surfaces were examined by scanning electron microscopy (SEM).

Needle-shaped specimens required for APT were fabricated by lift-outs and annular milled in a FEI Nova 200 focused ion beam/scanning electron microscope (FIB/SEM) [31,32]. The APT characterizations were performed in a local electrode atom probe (CAMEACA LEAP 4000X HR). The specimens were analyzed at 50 K in voltage mode, a pulse repetition rate of 200 kHz, a pulse fraction of 20%, and an evaporation detection rate of 0.5% atom per pulse. Imago Visualization and Analysis Software (IVAS) version 3.6.12 was used for creating the 3D

reconstructions and data analysis. The equivalent spherical particle radius, R , was determined by [33]:

$$R = \left(\frac{3V_p}{4\pi} \right)^{\frac{1}{3}}, \quad (1)$$

V_p is the volume of the particle as defined by the volume within the interface defined using

IVAS 3.6.12. The particle number density, N , were calculated from [33]:

$$N = \frac{N_p}{V}, \quad (2)$$

where N_p is the number of fully-contained plus 50% of the partially-contained particles within the volume, and V is the total volume of the APT reconstruction.

3. Results

3.1 Mechanical properties

The average hardness profiles measured along the transverse cross section of the welds in the AW, PWHT-550, and PWHT-600 conditions are shown in Fig. 1, which span the FZ, HAZ and BM. In the AW condition, the hardness profile shows a general softening in the FZ (~450 HV) as compared with the welding-unaffected BM (~605 HV), whereas a smooth transition decreasing from the BM to FZ can be observed in the HAZ. After PWHT for 30 min at 550 °C, the FZ shows a dramatic increase in hardness, reaching values of 580-600 HV, which is comparable to that of the HAZ and BM in this condition. Further increasing the PWHT temperature to 600 °C results in a slight decrease in hardness in all FZ, HAZ and BM, and the

hardness profile is approximately flat across the three regions, keeping at an average level of ~550 HV.

Room-temperature tensile tests were performed to further investigate the effects of welding and PWHT on the mechanical properties of the high-strength steels. The engineering stress-strain curves of the welds in the AW, PWHT-550, and PWHT-600 conditions are presented in Fig. 2, and the tensile curve of the original steel before welding (BW) is also included for comparison. Before welding, the original steel exhibits a yield strength of ~1670 MPa, an elongation-to-failure of ~9% and a reduction in area of ~40%, indicating a good combination of high strength and decent ductility [29]. In the AW condition, a considerable softening behavior is observed, with the yield strength decreasing to ~790 MPa. Meanwhile, the weld becomes progressively ductile, exhibiting a reduction in area of ~80%, double that in the BW condition. When the weld is heat-treated for 30 min at 550 °C, the yield strength is largely recovered, but the plastic ductility is completely lost. With an increasing PWHT temperature to 600 °C, both strength and ductility of the weld are mostly recovered, achieving a yield strength of ~1460 MPa, an ultimate strength of ~1520 MPa, a reduction in area of ~35%, and an elongation-to-failure of 6%.

The fracture surfaces of the steel in the BW, AW, PWHT-550, and PWHT-600 conditions are shown in Fig. 3. The original steel before welding shows a micro-void coalescence fracture mode with a lot of fine dimples (Fig. 3a), indicative of a ductile fracture. After welding and PWHT, all fractures were located in the FZ. In the AW condition, the necking becomes more prominent, and the dimples become more refined and numerous, as shown in Fig. 3b. However, the 550 °C PWHT changes the fracture mode from a ductile fracture to a brittle failure, with intergranular fracture observable in areas otherwise consisting of cleavage facets

(Fig. 3c). In comparison, the 600 °C PWHT results in a mixed fracture mode, mostly exhibiting fine dimples together with a small fraction of quasi-cleavage.

The above observations indicate that the welding process softens, instead of hardening and embrittlement, the nanoscale co-precipitation strengthened steel. The weld strength can be recovered by PWHT at 550-600 °C, while the weld ductility is very sensitive to the PWHT temperature. In this study, the 600 °C PWHT is a much better condition for the recovery of mechanical properties in terms of a combination of strength and ductility, as compared with that at 550 °C.

3.2 Grain structure characterization

The grain structures of the welds in the AW, PWHT-550, and PWHT-600 conditions were examined by SEM shown in Fig. 4a-e, and the corresponding prior-austenite grain sizes are summarized in Fig. 4f. No solidification cracks can be observed in both FZ and HAZ in all conditions, demonstrating that the nanoscale co-precipitation strengthened steel is weldable without any concerns for solidification cracking. In the AW condition, the BM and HAZ show a predominately lath martensite structure, with an average prior-austenite grain size of $\sim 10 \pm 3 \mu\text{m}$ and $\sim 11 \pm 4 \mu\text{m}$, respectively. In comparison, the FZ demonstrates freshly-formed untempered martensite with a coarse prior-austenite grain size of $\sim 32 \pm 7 \mu\text{m}$, which is considered to be a result of the rapid cooling of the weld pool. After the PWHT at 550 and 600 °C, the fresh martensite transforms to tempered martensite in the FZ, with no significant changes in the prior-austenite grain size (Fig. 4d and e).

To further characterize the phase structure of the welds, the FZ of the AW, PWHT-550, and PWHT-600 samples were examined by XRD, as illustrated in Fig. 5. In the AW and PWHT-

550 conditions, the XRD patterns show only the martensite peaks without any evidence of austenite phases. In contrast, after the 600 °C PWHT, the FZ consists mainly of martensite but with a small amount of austenite, as evidenced by the $\gamma(220)$ peak at the 2θ value of 74.5°, indicating the martensite-to-austenite reversion occurred during this treatment.

3.3 APT characterization

3.3.1 Before welding

The original steel before welding has been studied extensively by APT, and the previous data of sizes, number densities and compositions of nanoparticles will be referred to here for comparison with that in the welds in this study. Previous APT studies revealed the co-precipitation of high number densities of NiAl and Cu nanoparticles in the original steel after aging for 2 h at 550 °C. The average radii of NiAl and Cu nanoparticles are 1.7 ± 0.4 and 1.4 ± 0.4 nm, respectively, and the number densities of NiAl and Cu nanoparticles are 2.8×10^{24} and $5.2 \times 10^{23} \text{ m}^{-3}$, respectively [29]. In this condition, the NiAl nanoparticles contain 36.7 ± 0.4 at.% Ni, 26.9 ± 0.6 at.% Al, 8.3 ± 0.4 at.% Mn, 5.9 ± 0.2 at.% Cu and 22.2 ± 0.5 at.% Fe, whereas the Cu nanoparticles contain 31.4 ± 1.7 at.% Cu, 21.1 ± 1.5 at.% Ni, 12.6 ± 1.2 at.% Al, 6.0 ± 0.9 at.% Mn, and 28.9 ± 1.7 at.% Fe [29]. Hereafter, we refer to these values when discussing the precipitate microstructure and compositions of nanoparticles in the original steel before welding.

3.3.2 As-welded condition

The elemental distribution maps of the FZ in the AW condition is shown in Fig. 6, in which Ni, Al, Cu, and Mn atoms are depicted by red, cyan, green, and blue dots, respectively. In this region, all elements are homogeneously distributed, and no particular segregation can be

observable. The absence of NiAl and Cu nanoparticles in the FZ implies a complete precipitate dissolution during weld heating and no significant re-precipitation upon weld cooling. The Ni, Al, Mn, and Cu concentrations in the FZ solid solution were determined to be 5.09, 4.32, 2.85, and 1.28 at.%, respectively, which are quite similar to that in the bulk steel (4.74, 4.12, 3.04, and 1.31 at.%, respectively), further confirming that the pre-existing NiAl and Cu nanoparticles are almost completely dissolved into the matrix during the welding process.

Ni, Al, Mn, and Cu atom maps of the HAZ in the AW condition is displayed in Fig. 7a, which reveals co-segregation regions enriched in Ni, Al, Mn, and Cu. The 20 at.% (Ni + Al) concentration and 10 at.% Cu concentration isosurfaces were used to visualize the NiAl and Cu nanoparticles, respectively, as shown in Fig. 7b. The radii of NiAl and Cu nanoparticles are determined to be $\sim 2.4 \pm 0.5$ and $\sim 1.2 \pm 0.5$ nm, respectively, whereas their number densities are estimated to be $\sim 8.3 \times 10^{23}$ and $\sim 4.9 \times 10^{23} \text{ m}^{-3}$, respectively. As compared with the original steel before welding, the HAZ exhibits an increase in particle size and a decrease in number density. The proximity histograms of NiAl and Cu nanoparticles in the HAZ are displayed in Fig. 7c and d, respectively. The NiAl nanoparticles are enriched in Ni (34.9 ± 2.6 at.%) and Al (26.1 ± 0.4 at.%) but also contain a significant amount of Mn (10.9 ± 0.8 at.%) and Cu (14.4 ± 1.0 at.%). For the Cu nanoparticles, Cu (44.7 ± 6.1 at.%) are enriched in the particle center, whereas Ni, Al, Mn are segregated at the Cu particle-matrix interface.

3.3.3 Post-weld heat treated at 550 °C

The FZ solute distribution after the 550 °C PWHT is shown in Fig. 8a, and the isoconcentration surfaces of NiAl and Cu nanoparticles are displayed in Fig. 8b. Unlike the FZ in the AW condition which shows a supersaturation solid solution, spheroidal NiAl and Cu

nanoparticles are detected readily in the FZ of the PWHT550 sample. Both NiAl and Cu nanoparticles are extremely fine, with an average radius of $\sim 1.7 \pm 0.6$ and $\sim 1.3 \pm 0.3$ nm, respectively. The number density of NiAl nanoparticles is $\sim 2.5 \times 10^{24} \text{ m}^{-3}$, which is approximately fourfold that of Cu nanoparticles ($\sim 6.2 \times 10^{23} \text{ m}^{-3}$). The proximity histograms of NiAl and Cu nanoparticles in the FZ of the PWHT-550 sample are displayed in Fig. 8c and d, respectively. The NiAl nanoparticles contain mainly Ni ($36.3 \pm 2.3 \text{ at.}\%$) and Al ($26.6 \pm 1.8 \text{ at.}\%$) together with a small amount of Mn ($12.1 \pm 1.3 \text{ at.}\%$), whereas the Cu nanoparticles are enriched in Cu ($36.5 \pm 9.8 \text{ at.}\%$), Ni ($20.7 \pm 4.0 \text{ at.}\%$), and Al ($16.6 \pm 2.1 \text{ at.}\%$). It is interesting to find that the NiAl and Cu nanoparticles in the FZ of the PWHT-550 sample essentially have a similar size, number density and composition as that in the original steel before welding.

High number densities of NiAl and Cu nanoparticles are also detected in the HAZ of the PWHT-550 sample, as displayed in the atom maps in Fig. 9a and the isoconcentration surfaces in Fig. 9b. The NiAl particles show a slightly refined size ($\sim 2.2 \pm 0.6$ nm in average radius) and a larger number density ($\sim 1.2 \times 10^{24} \text{ m}^{-3}$), as compared with that in the AW condition, whereas the Cu nanoparticles have no significant changes, showing an average radius of $\sim 1.5 \pm 0.3$ nm and a number density of $\sim 4.4 \times 10^{23} \text{ m}^{-3}$. The proximity histograms of NiAl and Cu nanoparticles are displayed in Fig. 9c and d, respectively. The composition and solute partitioning behavior of NiAl and Cu nanoparticles are approximately unchanged, as compared with that in the AW condition.

3.3.4 Post-weld heat treated at 600 °C

APT atom distribution maps of the FZ after the 600 °C PWHT are illustrated in Fig. 10a, with the isoconcentration surfaces of NiAl and Cu nanoparticles in Fig. 10b. In this condition,

the NiAl and Cu nanoparticles have coarsened significantly, and the low number densities of coarse particles result in a small number of particles in similar volumes of analysis. The average radius and number density of NiAl nanoparticles are estimated to be $\sim 3.5 \pm 1.4$ nm and $\sim 3.6 \times 10^{23} \text{ m}^{-3}$, respectively, and that of Cu nanoparticles are estimated to be $\sim 2.2 \pm 1.4$ nm and $\sim 1 \times 10^{23} \text{ m}^{-3}$, respectively. The proximity histograms of NiAl and Cu nanoparticles are displayed in Fig. 10c and d, respectively. The NiAl nanoparticles become more enriched in Ni (42.2 ± 2.6 at.%) and Al (27.7 ± 1.1 at.%), whereas the Cu nanoparticles contain higher amount of Cu (61.1 ± 9.6 at.%), as compared with that in the PWHT-550 condition, indicating that both types of nanoparticles are moving closer to their equilibrium compositions.

APT analysis of the HAZ in the PWHT-600 condition, as shown in the atom maps in Fig. 11a and isoconcentration surfaces in Fig. 11b, also reveals coarse particles as observed for the FZ in the same condition. Due to the coarse nature of the particles, only a small number of particles can be observed in the volumes of analysis. The average radius and number density of NiAl nanoparticles are estimated to be $\sim 2.7 \pm 0.7$ nm and $\sim 5.6 \times 10^{23} \text{ m}^{-3}$, respectively. These coarse NiAl particles contain 44.2 ± 1.5 at.% Ni, 31.9 ± 0.9 at.% Al, and 8.5 ± 0.9 at.% Mn, as determined from the proximity histogram shown in Fig. 11c, which is similar to that of the FZ in the PWHT-600 condition. However, the size, number density, and composition of Cu nanoparticles cannot be quantitatively determined due to the small numbers of nanoparticles in the volume of analysis. Nevertheless, the Cu particles show a similar interfacial segregation of Ni and Al as observed for that in the FZ of the PWHT-600 sample (Fig. 11d).

4. Discussion

The above results show that the welding and PWHT can dramatically alter the microstructure and mechanical properties of the co-precipitation strengthened steel. In the following sections, the mechanism for the microstructural evolution and its correlation with the bulk mechanical properties will be discussed, aiming at elucidating the key factors governing the weld strength and ductility.

4.1 Microstructural evolution during welding and PWHT

The APT characterization of precipitate microstructure reveals complex solid-state transformations during welding and PWHT. For a quantitative understanding of the precipitate evolution, the particle size, number density, and composition of NiAl and Cu nanoparticles in the FZ at different states (i.e., BW, AW, PWHT-550, and PWHT-600) are summarized and compared in Fig. 12. In the BW condition, the steel is characterized by the co-precipitation of high number densities of ultrafine NiAl and Cu nanoparticles in a martensite matrix, which were precipitated from a supersaturated martensite matrix at 550 °C. Upon weld heating, the FZ matrix transforms from martensite to austenite and further to liquid, the temperature of which are predicted to be sufficiently high to completely dissolve all pre-existing NiAl and Cu nanoparticles. During weld cooling, the liquid transforms to coarse-grained austenite and then to martensite, the low-temperature phase. Unlike some carbide precipitates (such as NbC and TiC) which start to precipitate within the austenite region [34], NiAl and Cu nanoparticles do not precipitate from the austenite, avoiding the formation of coarse particles at high temperatures. This is because both Ni and Cu have a large solubility in austenite, and the concentrations of Ni and Cu in the studied steel (5 wt.% and 1.5 wt.%, respectively) lie within the solubility limit. As the matrix transforms from austenite to martensite, the solubility of Ni and Cu in the matrix

decreases dramatically, providing the possibility of NiAl and Cu precipitation at low temperatures. However, APT did not detect any NiAl or Cu particles in the as-welded FZ, and, in particular, the composition of the FZ solid solution is similar to the bulk composition. These observations clearly indicate that no particles are formed in the as-welded FZ, which has a high supersaturation of Cu and Ni in the AW condition. It is interesting to point out that the high supersaturation of the as-welded FZ solid solution, just like the solution-quenched one, offers precise control of the precipitate microstructure using subsequent treatments. After the 550 °C PWHT, high number densities of ultrafine NiAl and Cu nanoparticles re-precipitate out from the matrix in the FZ. It is interesting to note that the particle size (Fig. 12a), number density (Fig. 12b), and composition (Figs. 12c and d) of NiAl and Cu nanoparticles in the PWHT-550 condition are very similar to those in the original steel before welding, indicative of a fine-scale re-precipitation of NiAl and Cu nanoparticles in this condition. In comparison, the 600 °C PWHT results in a significant increase in size and a decrease in number density of nanoparticles, accompanied by the particle compositions approaching closer to their equilibrium ones, these implying that the 600 °C PWHT leads to coarse-scale re-precipitation of nanoparticles.

In addition to the precipitate morphology, the effect of welding and PWHT on the precipitation sequence of the co-precipitation strengthened steel was also explored. In the previous study on the original steel before welding, it has been found that at the initial stage of precipitation, NiAl nanoparticles, enriched in Ni and Al together with considerable amounts of Cu, first come out of the supersaturated solid solution; as the precipitation reaction continues, the first formation of NiAl particles leads to the rejection of Cu solutes outwards the NiAl particles, resulting in the heterogeneous precipitation of Cu nanoparticles adjacent to the NiAl nanoparticles [29]. Although the precipitate microstructures were not examined as a function of

PWHT time in this study, which is beyond the scope of this investigation, the size, number density, and spatial distribution of NiAl and Cu nanoparticles in the PWHT conditions can still give important information on the precipitation sequence. In the PWHT-550 condition (Fig. 8b), more than 60% NiAl particles are isolated ones without any Cu particles associated with them, whereas no isolated Cu particles are observable, and all Cu particles are located on the surface of NiAl particles. Moreover, the number density of NiAl nanoparticles ($\sim 2.5 \times 10^{24} \text{ m}^{-3}$) is approximately fourfold that of Cu nanoparticles ($\sim 6.2 \times 10^{23} \text{ m}^{-3}$). These observations suggest that NiAl particles precipitate out first and Cu particles form at a later stage of precipitation, consistent with the precipitation sequence observed in the thermally-aged steel [29]. Therefore, it appears that the welding and PWHT did not change the precipitation sequence of the NiAl/Cu co-precipitation strengthened steels.

Furthermore, the XRD pattern of the PWHT-600 sample reveals the formation of a small amount of reverted austenite, as evidenced by the γ peak in Fig. 5. It is known that the reversion of austenite is dependent strongly upon the annealing temperature, and reverted austenite has also been observed in many other martensitic steels when annealing at 600 °C or above, such as maraging steels [35-38] and medium-Mn steels [39,40]. To understand the temperature effect on the austenite reversion, thermodynamic calculations were performed to determine the equilibrium volume fraction of austenite of the studied steel at different temperatures, using the Thermo-Calc software together with the TCFE7 Fe-based database. The volume fractions of austenite at 550 and 600 °C are calculated to be $\sim 15\%$ and $\sim 25\%$, respectively, indicating that the formation of reverted austenite at 600 °C is not only kinetically enhanced but also thermodynamically favored, as compared with that at 550 °C.

4.2 Effects of welding and PWHT on the strength

The tensile stress–strain curves shown in Fig. 2 demonstrate that the welding and PWHT have a strong influence on the yield strength, which varies from ~1670 MPa in the BW condition, ~790 MPa in the AW condition, ~1610 MPa in the PWHT-550 condition, to ~1460 MPa in the PWHT-600 condition. The significant changes in yield strength can be explained mainly by the three microstructural changes that occurred during the welding and PWHT: (i) grain structure, (ii) precipitate microstructure, and (iii) austenite reversion. In the BW condition, the steel exhibits high number densities of ~3 nm NiAl and Cu nanoparticles ($\sim 2.8 \times 10^{24}$ and $5.2 \times 10^{23} \text{ m}^{-3}$, respectively), which are distributed uniformly in a martensite matrix with a fine prior-austenite grain size of $\sim 10 \pm 3 \text{ }\mu\text{m}$. The combination of precipitation strengthening, grain size strengthening, and martensite strengthening is considered to contribute majorly to the ~1670 MPa strength of the BW sample. After welding, all pre-existing NiAl and Cu nanoparticles are dissolved into the matrix, and the prior-austenite grain size increases by a factor of ~3. As the deformation is concentrated in the FZ, the complete loss of precipitation strengthening and reduction of grain-size strengthening result in a dramatic decrease of yield strength to ~790 MPa in the AW condition. The 550 °C PWHT produces fine-scale re-precipitation of ~3-nm NiAl and Cu nanoparticles ($\sim 2.5 \times 10^{24}$ and $\sim 6.2 \times 10^{23} \text{ m}^{-3}$ in number density, respectively), while the prior-austenite grain size ($\sim 30 \pm 6 \text{ }\mu\text{m}$) shows no apparent changes as compared with that in the AW condition ($\sim 32 \pm 7 \text{ }\mu\text{m}$). Thus, it can be stated that the re-precipitation of nanoparticles dramatically increases the strength from ~790 MPa in the AW condition to ~1610 MPa in the PWHT-550 condition, leading to a more than 90% recovery of strength relative to the original steel before welding. In comparison, the 600 °C PWHT leads to coarse-scale re-precipitation of

~5-nm NiAl and Cu nanoparticles ($\sim 2.5 \times 10^{24}$ and $6.2 \times 10^{23} \text{ m}^{-3}$ in number density, respectively), which gives rise to a slightly smaller strengthening effect, as compared with the 550 °C PWHT. In addition, although the prior-austenite grain size ($\sim 33 \pm 7 \text{ }\mu\text{m}$) does not show any significant differences after the 600 °C PWHT, the steel exhibits a small amount of reverted austenite, which generally leads to a decrease of strength [3]. As a consequence, the PWHT-600 sample exhibits a slightly lower strength ($\sim 1460 \text{ MPa}$) than the PWHT-550 sample ($\sim 1610 \text{ MPa}$), yielding an 85% recovery of strength. The 15% loss of strength relative to the original steel can be ascribed to the coarse-scale precipitation and coarse prior-austenite grain size together with the formation of a small amount of reverted austenite in the PWHT-600 condition.

4.3 Effects of welding and PWHT on the ductility

The tensile ductility of welds is critically important for structural steels for avoiding catastrophic failure in load-bearing applications. The above tensile tests demonstrate that the ductility is sensitive to the welding and PWHT. In the BW condition, the ultra-high strength steel exhibits a good ductility of 9% elongation-to-failure and more than 40% reduction in area at the necking cross-section. After welding, the deformation is essentially concentrated in the FZ. The steel becomes softer and more ductile ($\sim 80\%$ reduction in area), rather than hard and brittle as often observed for many high-carbon steels. It is known that carbon is one of the most important alloying elements affecting the steel weldability, and high-carbon steels tend to suffer from solidification cracking in the welds because of the formation of hard and brittle martensite [1,41]. In contrast, the ultra-high strength steel strengthened by NiAl and Cu nanoparticles contains only a small amount of carbon ($\sim 0.05 \text{ wt.}\%$). Thus, the low-carbon matrix would transform to a soft martensite, rather than a hard and brittle martensite, after weld cooling, which facilitates the

formation of crack-free welds with a good ductility. The 550 °C PWHT results in a complete loss of ductility, whereas the 600 °C PWHT leads to a most recovery of ductility, achieving a reduction in area of ~35% and an elongation-to-failure of ~6%. It appears that the weld ductility has a strong dependence on the PWHT temperature, similar as the temper embrittlement phenomenon of quenched-and-tempered steels [42-46]. In general, the mechanism for temper embrittlement and de-embrittlement of steels can be related to the following factors: (1) grain size, (2) precipitate microstructure, (3) grain-boundary segregation, and (4) reversion of austenite [5,42-46]. In this study, the difference of ductility between the PWHT-550 and PWHT-600 conditions will be discussed from the above four aspects. First, there is no significant difference in the prior-austenite grain size between the PWHT-550 ($30 \pm 6 \mu\text{m}$) and PWHT-600 ($33 \pm 7 \mu\text{m}$) conditions, so the grain size cannot be a dominant factor causing the difference in ductility between the two conditions. Second, the PWHT-600 sample shows coarser and more sparsely distributed precipitates than the PWHT-550 sample, as illustrated in Fig. 12a. The particle coarsening would result in a reduced precipitation strengthening response and hence a slightly lower yield strength, which may help the ductility recovery. Nevertheless, it is unlikely that the fine-scale re-precipitation would be a major factor deteriorating the weld ductility, because the original steel, which also exhibits fine precipitates (~1.5 nm) similar to the PWHT-550 sample (~1.4 nm), shows a good ductility with an elongation of ~9%. Third, since the intergranular fracture occurred in the PWHT-550 condition but not in the PWHT-600 condition, the grain-boundary segregation of the PWHT-550 sample were examined by APT. Figure 13 shows atom maps of Ni, Al, Cu, Mn, Mo, W, C and P in a region across a ferrite-austenite boundary. Although nanoparticles enriched in Ni, Al, and Cu can be clearly visible at the grain boundary, it is unlikely that the grain-boundary precipitation of nanoparticles would be a principle factor

causing the intergranular fracture, because these particles have ultra-fine sizes similar to those in the grain interior, instead of coarse ones. From the atom maps, it is interesting to find the grain-boundary segregation of Mn, Mo, W, C, and P. Previous studies suggest that Mo, W and C are grain-boundary strengtheners [47], whereas the segregation of Mn and P generally cause embrittlement by decreasing the grain-boundary cohesion in many steels [47-52]. Therefore, the grain-boundary segregation of Mn and P could be a main reason for the 550 °C embrittlement of the coarse-grained weld. It is known that, in general, the equilibrium segregation of an element decreases with increasing temperature [5]. Thus, the 600 °C PWHT is expected to reduce the grain-boundary segregation of both Mn and P, thereby avoiding the grain-boundary fracture. Fourth, XRD reveals that the 600 °C PWHT leads to the reversion of austenite, whereas the matrix appears to be dominantly martensitic after PWHT for 30 min at 550 °C. Previous studies suggest that the reverted austenite is possible to act as a soft barrier against incoming cracks or stress-strain localizations from the martensite, preventing cracks from percolating from one grain into another [51]. Therefore, the reversion of austenite could be another important contributor to the improved ductility in the PWHT-600 condition. Based on all the above discussion, it appears that the reduced grain-boundary segregation of Mn and P and the reversion of austenite could be two important reasons for the enhanced ductility of the PWHT-600 sample as compared with the PWHT-550 one, whereas the grain size and precipitate microstructure play no or a minor role in causing the ductility difference in the two conditions.

5. Summary

Based on the study of the effects of welding and PWHT on the microstructure and mechanical properties of the ultra-high strength steel strengthened by NiAl and Cu nanoparticles with sizes in the range of 1-10 nm, the following conclusions are drawn:

1. APT reveals that the welding process dissolves all pre-existing nanoparticles, resulting in a supersaturated FZ solid solution similar to the solution-quenched microstructure except for the coarse prior-austenite grain size. The 550 °C PWHT leads to fine-scale re-precipitation of NiAl and Cu co-precipitates with high number densities and ultra-fine sizes, similar to that in the original steel before welding, whereas the 600 °C PWHT gives rise to coarse-scale re-precipitation of nanoparticles together with the formation of a small amount of reverted austenite.
2. The welding and PWHT have a strong influence on the steel strength, which can be explained mainly by the grain structure change, precipitate microstructure evolution, and austenite reversion. The welding process results in a complete loss of precipitation strengthening and a reduction of grain-size strengthening, both of which greatly soften the steel. The 550 °C PWHT leads to a large recovery of strength mainly due to the fine-scale re-precipitation of NiAl and Cu co-precipitates, whereas the 600 °C PWHT gives a major part of the strength recovery. The loss of strength in the PWHT-600 condition can be ascribed to the coarse-scale precipitation and coarse prior-austenite grain size together with the formation of a small amount of reverted austenite.
3. In the AW state, the steel becomes soft and ductile without any cracks, rather than hard and brittle as often observed for many high-carbon steels. However, the 550 °C PWHT results in a complete loss of ductility with intergranular failure, and APT reveals that the grain-boundary segregation of Mn and P could be the main reason for this embrittlement.

In comparison, the 600 °C PWHT leads to a large recovery of ductility, which can be ascribed mainly to a possible reduction of grain-boundary segregation and reversion of austenite.

4. The PWHT has been approved to be an effective method for the recovery of mechanical properties of nanoscale co-precipitation strengthened steels, the degree of which is dependent strongly upon the PWHT temperature.

Acknowledgments

This research was supported by the General Research Fund (account No. CityU11205515) and the Collaborative Research Fund (account No. C1027-14E GRF) from the Research Grant Council, Hong Kong. Atom probe tomography (J.D.P. and W.G.) was conducted at ORNL's Center for Nanophase Materials Sciences (CNMS), which is a U.S. DOE Office of Science User Facility.

References

- [1] R.W. Cahn, P. Haasen, Physical metallurgy (4th edn), North-Holland, Amsterdam, 1996.
- [2] M.E. Fine, S. Vaynman, D. Isheim, Y.W. Chung, S.P. Bhat, C.H. Hahin, A new paradigm for designing high-fracture-energy steels, *Metall. Mater. Trans. A* 41 (2010) 3318.
- [3] A. Saha, G.B. Olson, Computer-aided design of transformation toughened blast resistant naval hull steels: Part I, *J. Comput. Aided Mater. Des.* 14 (2007) 177.
- [4] M. Kapoor, D. Isheim, G. Ghosh, S. Vaynman, M.E. Fine, Y.W. Chung, Aging characteristics and mechanical properties of 1600MPa body-centered cubic Cu and B2-NiAl precipitation-strengthened ferritic steel, *Acta Mater.* 73 (2014) 56.
- [5] M. Kapoor, D. Isheim, S. Vaynman, M.E. Fine, Y.W. Chung, Effects of increased alloying element content on NiAl-type precipitate formation, loading rate sensitivity, and ductility of Cu-and NiAl-precipitation-strengthened ferritic steels, *Acta Mater.* 104 (2016) 166.

- [6] S. Huang, Y. Gao, K. An, L. Zheng, W. Wu, Z. Teng, P.K. Liaw, Deformation mechanisms in a precipitation-strengthened ferritic superalloy revealed by in situ neutron diffraction studies at elevated temperatures, *Acta Mater.* 83 (2015) 137.
- [7] H.W. Yen, P.Y. Chen, C.Y. Huang, J.R. Yang, Interphase precipitation of nanometer-sized carbides in a titanium-molybdenum-bearing low-carbon steel, *Acta Mater.* 59 (2011) 6264.
- [8] G. Trotter, G. Rayner, I. Baker, P.R. Munroe, Accelerated precipitation in the AFA stainless steel Fe-20Cr-30Ni-2Nb-5Al via cold working, *Intermetallics* 53 (2014) 120.
- [9] S.R. Goodman, S.S. Brenner, J.R. Low, An FIM-atom probe study of the precipitation of copper from Iron-1.4 at. pct copper. Part I: field-ion microscopy, *Metall. Trans.* 4 (1973) 2363.
- [10] D. Isheim, M.S. Gagliano, M.E. Fine, D.N. Seidman, Interfacial segregation at Cu-rich precipitates in a high-strength low-carbon steel studied on a sub-nanometer scale, *Acta Mater.* 54 (2006) 841.
- [11] R.P. Kolli, D.N. Seidman, The temporal evolution of the decomposition of a concentrated multicomponent Fe-Cu-based steel, *Acta Mater.* 56 (2008) 2073.
- [12] Z.B. Jiao, J.H. Luan, Z.W. Zhang, M.K. Miller, W.B. Ma, C.T. Liu, Synergistic effects of Cu and Ni on nanoscale precipitation and mechanical properties of high-strength steels, *Acta Mater.* 61 (2013) 5996.
- [13] R. Taillard, A. Pineau, Room temperature tensile properties of Fe-19wt.% Cr alloys precipitation hardened by the intermetallic compound NiAl, *Mater. Sci. Eng.* 56 (1982) 219.
- [14] Z.K. Teng, M.K. Miller, G. Ghosh, C.T. Liu, S. Huang, K.F. Russell, M.E. Fine, P.K. Liaw, Characterization of nanoscale NiAl-type precipitates in a ferritic steel by electron microscopy and atom probe tomography, *Scr. Mater.* 63 (2010) 61.
- [15] N.Q. Vo, C.H. Liebscher, M.J. Rawlings, M. Asta, D.C. Dunand, Creep properties and microstructure of a precipitation-strengthened ferritic Fe-Al-Ni-Cr alloy, *Acta Mater.* 71 (2014) 89.
- [16] Z.B. Jiao, J.H. Luan, M.K. Miller, C.Y. Yu, C.T. Liu, Effects of Mn partitioning on nanoscale precipitation and mechanical properties of ferritic steels strengthened by NiAl nanoparticles, *Acta Mater.* 84 (2015) 283.

- [17] Z.B. Jiao, J.H. Luan, M.K. Miller, C.Y. Yu, Y. Liu, C.T. Liu, Precipitate transformation from NiAl-type to Ni₂AlMn-type and its influence on the mechanical properties of high-strength steels, *Acta Mater.* 110 (2016) 31.
- [18] A.E. Amer, M.Y. Koo, K.H. Lee, S.H. Kim, S.H. Hong, Effect of welding heat input on microstructure and mechanical properties of simulated HAZ in Cu containing microalloyed steel, *J. Mater. Sci.* 45 (2010) 1248.
- [19] X.Yu, J.L. Caron, S.S. Babu, J.C. Lippold, D. Isheim, D.N. Seidman, Characterization of microstructural strengthening in the heat-affected zone of a blast-resistant naval steel, *Acta Mater.* 58 (2010) 5596.
- [20] J.L. Caron, S.S. Babu, J.C. Lippold, Weldability evaluation of a Cu-bearing high-strength blast-resistant steel, *Metall. Mater. Trans. A* 42 (2011) 4032.
- [21] A.H. Hunter, J.D. Farren, J.N. DuPont, D.N. Seidman, An atom-probe tomographic study of arc welds in a multi-component high-strength low-alloy steel, *Metall. Mater. Trans. A* 44 (2013) 1741.
- [22] X. Yu, J.L. Caron, S.S. Babu, J.C. Lippold, D. Isheim, D.N. Seidman, Strength recovery in a high-strength steel during multiple weld thermal simulations, *Metall. Mater. Trans. A* 42 (2011) 3669.
- [23] J.T. Bono, J.N. DuPont, D. Jain, S.I. Baik, D. N. Seidman, Investigation of Strength Recovery in Welds of NUCu-140 Steel Through Multipass Welding and Isothermal Post-Weld Heat Treatments, *Metall. Mater. Trans. A* 46 (2015) 5158.
- [24] M.D. Mulholland, D.N. Seidman, Nanoscale co-precipitation and mechanical properties of a high-strength low-carbon steel, *Acta Mater.* 59 (2011) 1881.
- [25] G. Song, Z. Sun, L. Li, X. Xu, M. Rawlings, C.H. Liebscher, B. Clausen, J. Poplawsky, D.N. Leonard, S. Huang, Z. Teng, C.T. Liu, M.D. Asta, Y. Gao, D.C. Dunand, G. Ghosh, M. Chen, M.E. Fine, P.K. Liaw, Ferritic Alloys with Extreme Creep Resistance via Coherent Hierarchical Precipitates, *Sci. Rep.* 5 (2015) 16327.
- [26] C.H. Liebscher, V.R. Radmilović, U. Dahmen, N.Q. Vo, D.C. Dunand, M. Asta, G. Ghosh, A hierarchical microstructure due to chemical ordering in the bcc lattice: Early stages of formation in a ferritic Fe–Al–Cr–Ni–Ti alloy, *Acta Mater.* 92 (2015) 220.

- [27] J. Millán, S. Sandlöbes, A. Al-Zubi, T. Hickel, P. Choi, J. Neugebauer, D. Ponge, D. Raabe, Designing Heusler nanoprecipitates by elastic misfit stabilization in Fe–Mn maraging steels, *Acta Mater.* 76 (2014) 94.
- [28] Z.W. Zhang, C.T. Liu, M.K. Miller, X.L. Wang, Y.R. Wen, T. Fujita, A. Hirata, M.W. Chen, G. Chen, B.A. Chin, A nanoscale co-precipitation approach for property enhancement of Fe-base alloys, *Sci. Rep.* 3 (2013) 1327.
- [29] Z.B. Jiao, J.H. Luan, M.K. Miller, C.T. Liu, Precipitation mechanism and mechanical properties of an ultra-high strength steel hardened by nanoscale NiAl and Cu particles, *Acta Mater.* 97 (2015) 58.
- [30] Z.B. Jiao, J.H. Luan, M.K. Miller, C.Y. Yu, C.T. Liu, Group precipitation and age hardening of nanostructured Fe-based alloys with ultra-high strengths, *Sci. Rep.* 6 (2016) 21364.
- [31] K. Thompson, D. Lawrence, D. J. Larson, J. D. Olson, T. F. Kelly and B. Gorman, In situ site-specific specimen preparation for atom probe tomography, *Ultramicroscopy* 107 (2007) 131.
- [32] G. Wei, A.G. David, D.T. Julie, H. Daniel, A.Y. George, D.P. Jonathan, An atom probe perspective on phase separation and precipitation in duplex stainless steels, *Nanotechnology* 27 (2016) 254004.
- [33] M.K. Miller, R.G. Forbes, *Atom-Probe Tomography: The Local Electrode Atom Probe*, Springer, New York, 2014.
- [34] M. Charleux, W.J. Poole, M. Militzer, A. Deschamps, Precipitation behavior and its effect on strengthening of an HSLA-Nb/Ti steel, *Metall. Mater. Trans. A* 32 (2001) 1635.
- [35] U.K. Viswanathan, G.K. Dey, V. Sethumadhavan, Effects of austenite reversion during overageing on the mechanical properties of 18 Ni (350) maraging steel, *Mater. Sci. Eng. A* 398 (2005) 367.
- [36] M. Farooque, H. Ayub, A.U. Haq, A.Q. Khan, The formation of reverted austenite in 18% Ni 350 grade maraging steel, *J. Mater. Sci.* 33 (1998) 2927.
- [37] Z. Guo, W. Sha, D. Li, Quantification of phase transformation kinetics of 18 wt.% Ni C250 maraging steel, *Mater. Sci. Eng. A* 373 (2004) 10.

- [38] R. Schnitzer, R. Radis, M. Nöhrer, M. Schober, R. Hochfellner, S. Zinner, E. Povoden-Karadeniz, E. Kozeschnik, H. Leitner, Reverted austenite in PH 13-8 Mo maraging steels, *Mater. Chem. Phys.* 122 (2010) 138.
- [39] D. Raabe, S. Sandlöbes, J. Millán, D. Ponge, H. Assadi, M. Herbig, P. P. Choi, Segregation engineering enables nanoscale martensite to austenite phase transformation at grain boundaries: A pathway to ductile martensite, *Acta Mater.* 61 (2013) 6132.
- [40] M.M. Wang, C.C. Tasan, D. Ponge, D. Raabe, Spectral TRIP enables ductile 1.1 GPa martensite, *Acta Mater.* 111 (2016) 262.
- [41] N. Yurioka, Physical metallurgy of steel weldability, *ISIJ Int.* 41 (2001) 566.
- [42] M. Guttman, Equilibrium segregation in a ternary solution: A model for temper embrittlement, *Surf. Sci.* 53 (1975). 213.
- [43] I. Olefjord, Temper embrittlement, *Int. Metals Rev.* 23 (1978) 149.
- [44] W.R. Tyson, Kinetics of temper embrittlement, *Acta Metall.* 26 (1978) 1471.
- [45] R.M. Horn, R.O. Ritchie, Mechanisms of tempered martensite embrittlement in low alloy steels, *Metall. Trans. A*, 9 (1978) 1039.
- [46] S.K. Dhua, D. Mukerjee, D.S. Sarma, Influence of thermomechanical treatments on the microstructure and mechanical properties of HSLA-100 steel plates, *Metall. Mater. Trans. A* 34 (2003) 241.
- [47] W.T. Geng, A.J. Freeman, G.B. Olson, Influence of alloying additions on grain boundary cohesion of transition metals: first-principles determination and its phenomenological extension, *Phys. Rev. B* 63 (2001) 165415.
- [48] C.L. Briant, S.K. Banerji, Phosphorus induced 350° C embrittlement in an ultra high strength steel, *Metall. Trans. A* 10 (1979) 123.
- [49] A. Salemi, A. Abdollah-Zadeh, The effect of tempering temperature on the mechanical properties and fracture morphology of a NiCrMoV steel, *Mater. Charact.* 59 (2008) 484.
- [50] R.P. Messmer, C.L. Briant, The role of chemical bonding in grain boundary embrittlement, *Acta Metall.* 30 (1980) 457.
- [51] M. Kuzmina, D. Ponge, D. Raabe, Grain boundary segregation engineering and austenite reversion turn embrittlement into toughness: example of a 9wt.% medium Mn steel, *Acta Mater.* 86 (2015) 182.

- [52] F. Moszner, S.S.A. Gerstl, P.J. Uggowitzer, J.F. Löffler, Atomic-scale characterization of prior austenite grain boundaries in Fe–Mn-based maraging steel using site-specific atom probe tomography, *Acta Mater.* 73 (2014) 215.

Fig. 1 Hardness profiles across the fusion zone, heat-affected zone and base metal in the as-welded and post-weld heat treated conditions.

Fig. 2 Tensile stress-strain curves of the nanoscale co-precipitation strengthened steels in different conditions: before welding, as-welded, and post-weld heat treated conditions.

Fig. 3 Fracture surfaces of the nanoscale co-precipitation strengthened steel before welding and in the as-welded and post-weld heat treated conditions.

Fig. 4 Grain structures of the nanoscale co-precipitation strengthened steel in different conditions: (a) base metal, as-welded, (b) heat-affected zone, as-welded, (c) fusion zone, as-welded, (d)

fusion zone, post-weld heat treated at 550 °C, (e) fusion zone, post-weld heat treated at 600 °C, and (f) prior-austenite grain size in these conditions.

Fig. 5 XRD patterns of the nanoscale co-precipitation strengthened steel in the as-weld and post-weld heat treated conditions.

Fig. 6 Atom maps of Ni, Al, Cu and Mn of the fusion zone in the as-welded condition. All elements exhibit a homogeneous distribution.

Fig. 7 APT characterization of the heat-affected zone in the as-welded condition: (a) atom maps of Ni, Al, Cu and Mn, (b) isoconcentration surfaces of nanoparticles, and proximity histograms of (c) NiAl and (d) Cu nanoparticles.

Fig. 8 APT characterization of the fusion zone in the 550 °C PWHT condition: (a) atom maps of Ni, Al, Cu and Mn, (b) isoconcentration surfaces of nanoparticles, and proximity histograms of (c) NiAl and (d) Cu nanoparticles.

Fig. 9 APT characterization of the heat-affected zone in the 550 °C PWHT condition: (a) atom maps of Ni, Al, Cu and Mn, (b) isoconcentration surfaces of nanoparticles, and proximity histograms of (c) NiAl and (d) Cu nanoparticles.

Fig. 10 APT characterization of the fusion zone in the 600 °C PWHT condition: (a) atom maps of Ni, Al, Cu and Mn, (b) isoconcentration surfaces of nanoparticles, and proximity histograms of (c) NiAl and (d) Cu nanoparticles.

Fig. 11 APT characterization of the heat-affected zone in the 600 °C PWHT condition: (a) atom maps of Ni, Al, Cu and Mn, (b) isoconcentration surfaces of nanoparticles, and proximity histograms of (c) NiAl and (d) Cu nanoparticles.

Fig. 12 (a) Particle radius and (b) number density of nanoparticles in the steel before welding and in the as-welded and post-weld heat treated conditions, and compositions of (c) NiAl and (d) Cu nanoparticles in the steel before welding and in the post-weld heat treated conditions.

Fig. 13 Atom maps of Ni, Al, Cu, Mn, Mo, W, C, and P across a ferrite-austenite boundary of the fusion zone in the 550 °C PWHT condition.