2.6 FRICTION STIR WELDING OF ODS STEELS AND ADVANCED FERRITIC STRUCTURAL STEELS

— Z. Feng, X. Yu, W. Tang, D. Hoelzer, and L.T. Tan (Oak Ridge National Laboratory)

OBJECTIVE

This project aims to address the critical technology gap of joining oxide dispersion strengthened (ODS) steels, nanostructured ferritic alloys (NFAs), reduced-activation ferritic/martensitic (RAFM) steels, and dissimilar metal joining of ODS/NFAs and RAFM steels through friction stir welding technology. The research focuses on understanding the stability of the strengthening phases in the weld region, and the bonding mechanisms between dissimilar structural steels as a function of FSW process conditions.

Specific project objectives are (i) developing the process parameter space to consistently produce defect-free welds of the same and dissimilar metals, (ii) developing the knowledge base and practical applicable approaches to tailor and optimize the microstructure features in the weld to match the properties of the base metal through process innovation, and (iii) producing representative weld joints and welded components to support future testing and evaluation at high temperature and irradiation environments in collaboration with JAEA and other international teams.

SUMMARY

Our research during this reporting period focused on (1) understanding the effects of post-weld heat treatment (PWHT) on the microstructure and property variations in friction stir welds of RAFM steels, (2) developing the PWHT schedule to homogenize and restore the weld properties to match these of base metal, and (3) understanding the effect of the extreme thermal and deformation conditions associated with FSW on the nano oxide strengthening particles in MA956 ODS alloy. Major findings included the development of a PWHT schedule to produce uniform microstructure and a homogeneous hardness distribution in the weld region that is identical to base metal. Initial atom probe maps of FSWed MA956 revealed complete dissolution of the nano oxide particles, which has major implications on the fundamental understanding the stability of nano-oxide particles under extreme thermal-mechanical process conditions, as well as potential solutions to restore the creep strength of the weld region.

PROGRESS AND STATUS

Post-Weld Heat Treatment Study of RAFM FSW

Previously, friction stir welding trials were carried out on two RAFM steels (EUROFER97 and experimental RAFM steel with a composition of Fe-9Cr-1.48W-0.13Ta-0.09C). The as-welded microstructure within stir zone (SZ) and heat-affected zone (HAZ) are distinctively different (Figure 1 (a)-(c)), which led to a highly inhomogeneous distribution of mechanical property, e.g., hardness, within the welds. Due to high hardenability of RAFM steels, the stir zone was predominately martensite with its characteristic higher hardness. Softening was found in the HAZ due to over-tempering of the hardened phases and/or the formation of pre-eutectoid ferrite in the inter-critical region. Post-weld heat treatment (PWHT), which is commonly used for such type of steels, was considered as an option to restore the microstructure and properties in the weld region to these of the base metal. Results from the following three representative PWHT cases are presented.

- Case 1: Tempering at 760°C for 0.5hr followed by air cooling
- Case 2: Tempering at 800°C for 0.5hr followed by air cooling
- Case 3: Normalizing at 980°C for 0.5hr and quench, followed by tempering at 760°C for 0.5hr with air-cooling.

Case 3 represents the typical heat treatment conditions applied to a RAFM steel in producing the optimal high-temperature properties. In the other two cases, PWHT temperature was progressively increased in an attempt to temper the martensite in the stir zone but without re-austenizing the material.

Microstructures of PWHT specimens within the stir zone (SZ), thermo-mechanically affected zone (TMAZ), and HAZ were examined under optical microscope. Hardness mapping was also performed on the cross section of the welds using LM100AT micro-hardness tester, in order to reveal the effect of PWHT on the mechanical property. Sample surface was grinded, polished, and etched with a mixture of 30ml HCl and 10ml HNO_3, to reveal the microstructure and to exam the welding defects.

The microstructure and microhardness of FSWed EUROFER92 are shown in Figures 1 and 2 respectively. The effects of PWHT on the experimental RAFM steel are shown in Figures 3 and 4. First of all, the as-welded microstructures and hardnesses are drastically different from the base metal. Tempering only PWHT (at least for the time duration studied in this performance period) was not able to homogenize the microstructure in the stir zone and the HAZ. On the other hand, applying the heat treatment conditions of the base metal restored the microstructure and hardness to these of the base metal (with some minor variations in EUROFER92 weld). This suggested the advantage of FSW – since no additional materials (filler metal) is used in FSW, the weld region has essentially the same chemistry as the base metal so that it is possible to restore the microstructure and properties of the weld region to these of the base metal through proper heat treatment. This is the primary goal of the project.

In the next few months, additional heat treatment studies and FSW process conditions will be further studied, with the objective of developing effective approaches to tailor and optimize the microstructure features in the weld to match the properties of the base metal.

The restoration of a homogeneous microstructure and microhardness through PWHT forms a sound foundation for further testing the high-temperature mechanical properties of the FSWed RAFM steel and comparing with the base metal properties. Mechanical property testing is planned in the next few months.

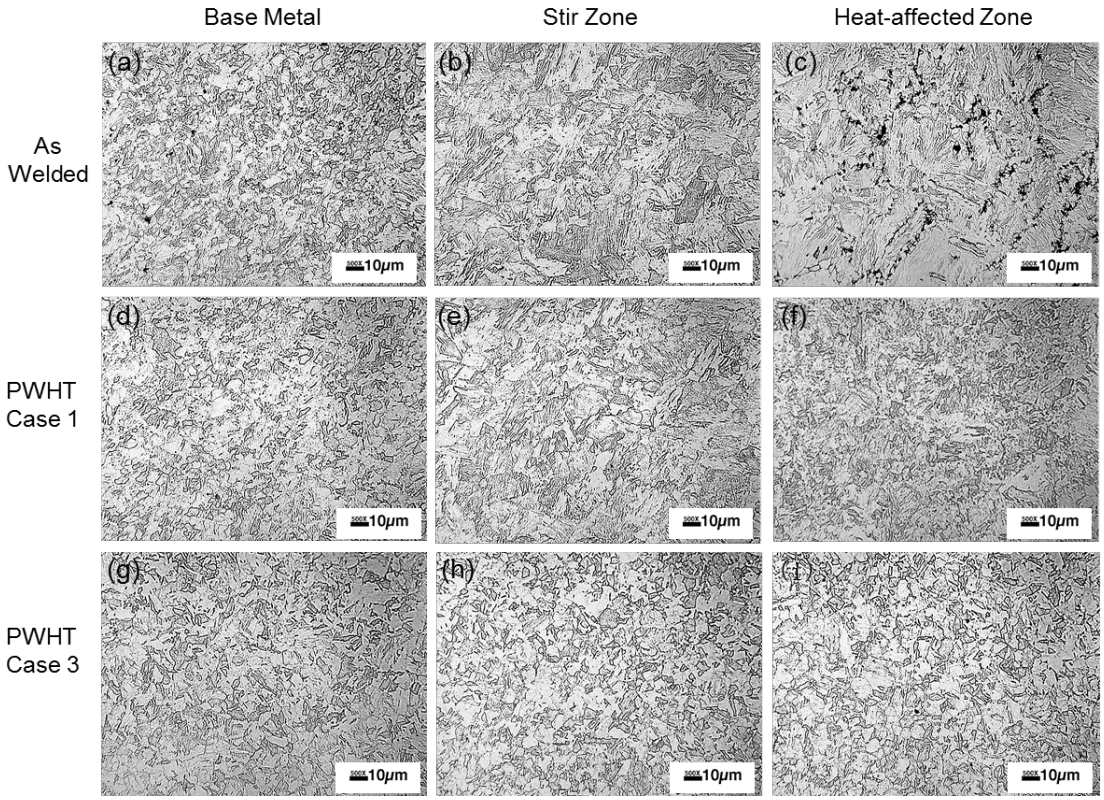

Figure 1. Optical micrographs of the as-welded (a)-(c) and PWHTed EUROFER 97 samples under conditions of Case 1 (d)-(f) and Case (3) (g)-(i) within base metal, stir zone and heat-affected zone, respectively.

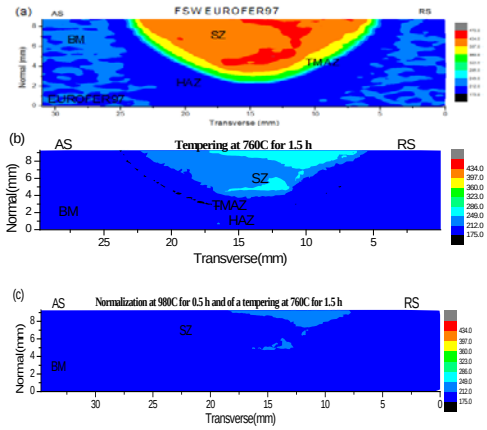

Figure 2. Hardness mappings of the FSW EUROFER97 steel as-welded (a), PWHT Case 1 (b) and 3 (c)

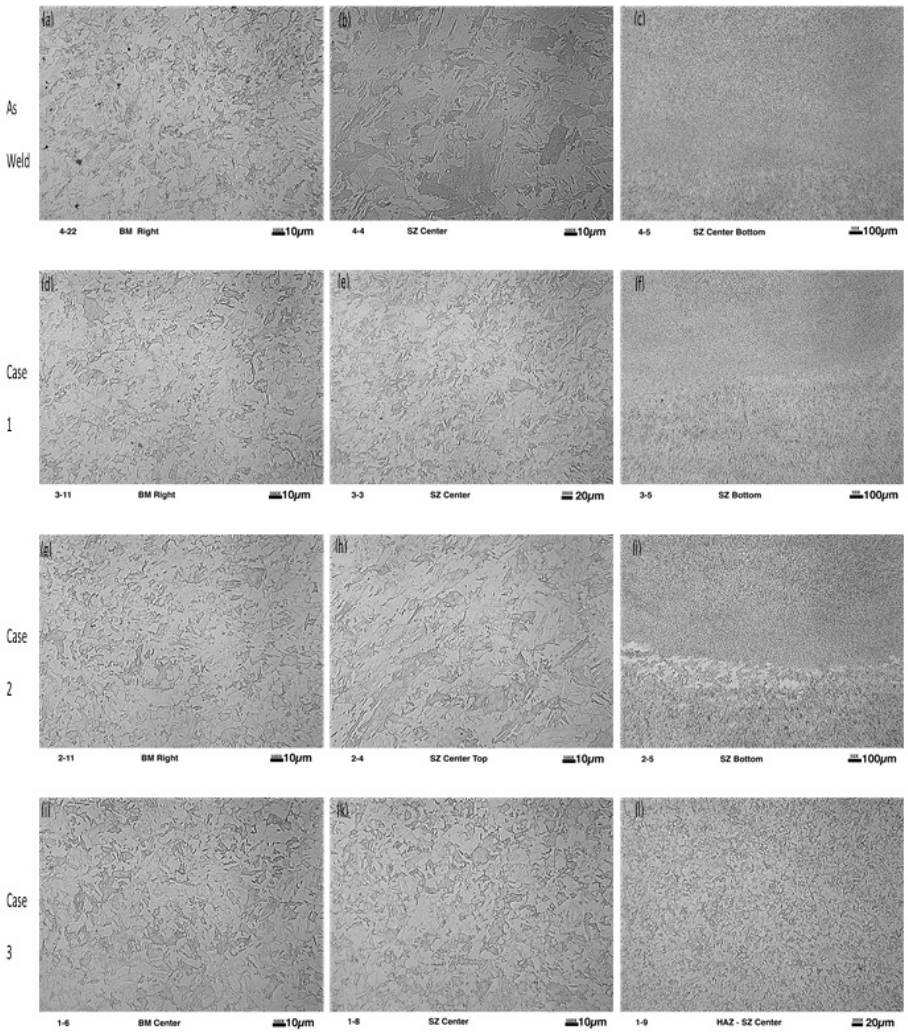

Figure 3. Optical micrographs of the as-welded (a)-(c) and PWHTed experimental RAFM steel samples under conditions of Case 1 (d)-(f) , Case (2) (g)-(i) and Case 3 (j)-(l) within base metal, stir zone respectively.

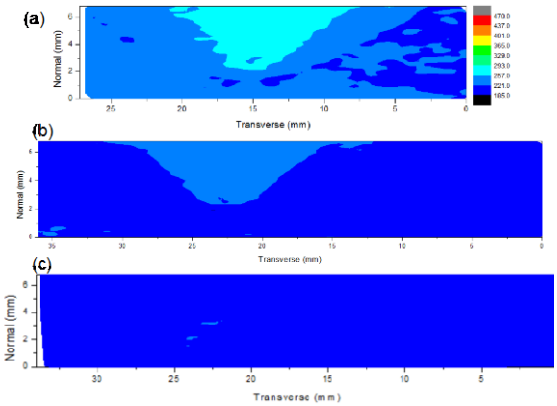

Figure 4. Hardness mappings of the FSWed experimental RAFM steels after PWHT in Cases 1-3 (a)-(c)

Additional FSW welds on RAFM steel for planned mechanical property testing

Additional friction stir welds were made on RAFM steel in this period, using the welding parameters developed which results in defect free welds. These welds are planned for further studying of post weld heat treatment and mechanical property testing. Due to the limited availability of the RAFM steel, the mechanical testing will be on a limited scoping study.

Figure 5 shows the apparent of the FSW made. Note the surface was painted for DIC measurement of stress/strain distribution during welding. Also shown in the figure are the thermocouples used to calibrate the temperature field measurement by Infrared thermography. The strain field during the friction stir welding process is being analyzed and will be corroborated with the temperature field measurement to further our understanding of the FSW process in joining creep resistance materials.

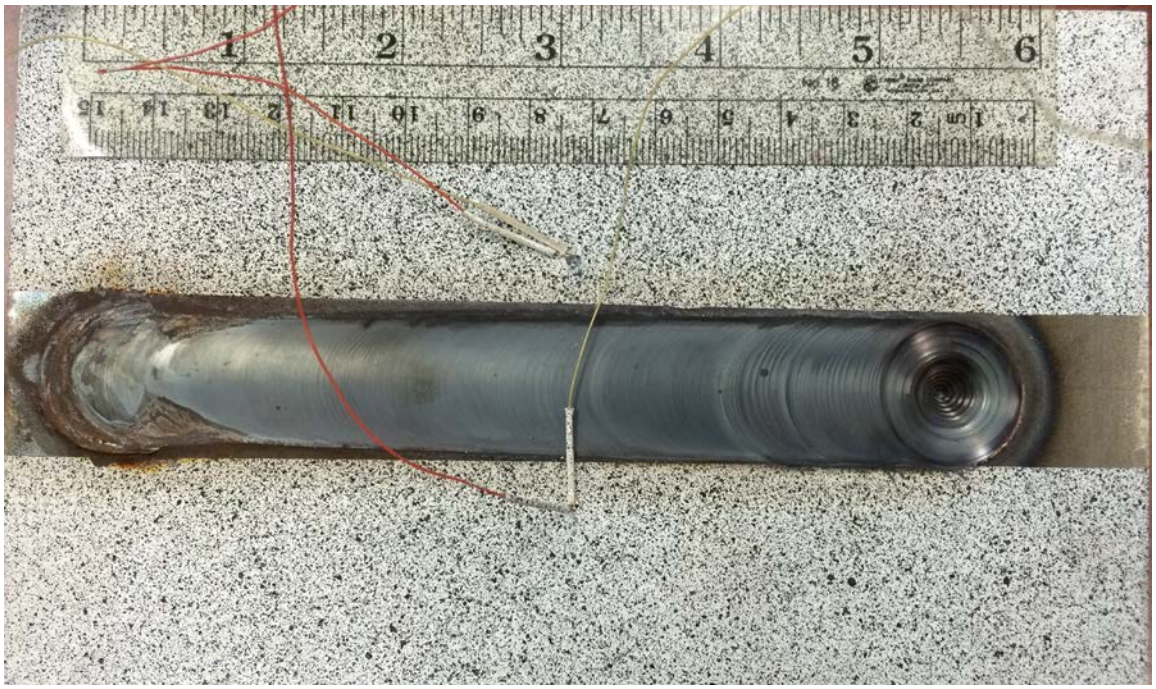

Figure 5. Surface appearance of RAFM FSW.

Characterization of Nano Oxide Particles in MA956 ODS Alloy Friction Stir Weld

As reported previously, defect-free FSW between MA956 ODS alloy and RAFM steels were successfully produced in this project. However, the stir region of MA956 ODS alloy showed considerable reduction of microhardness compared to the base metal. Systematic investigations of the underlying microstructure changes are on-going. Initial atom probe tomography (APT) analysis revealed some very interesting results, as shown in Figure 6 and Figure 7. In Figure 6, the clusters of nano-sized oxide particles are clearly observed in the base metal of MA956. The particles, with size between 10-50 nm, are enriched with in O, Y, and Al. However, as shown in Figure 7, these particles, at least for the regions examined so far, are near completely resolved (or re-solutionized) in the stir zone, as evidenced by no spherical oxide found. It should be noted the absence of Y element in Figure 7. Such absence will be further investigated in the next round of experiment. It is worth noting the absent of Yttrium element in the matrix, which is difficult to comprehend at this moment. Such observations were very different from recent work by others where FSW tends to coarsen the nano particles or nano-features in different ODS alloys. Unlike the coarsening case, the resolution of nano particles is significant in that it offers the possibility to form the

nano-particles through appropriate post weld heat treatment. This aspect will be investigated in the next few months.

It is important to recognize that the stability of the nano-particles is dependent on the chemistry and nature of these particles. Other type of nano-particles or nano-clusters such as those in 14YWT ODS alloys which do not have Al may have different stability during FSW. This aspect is being investigated.

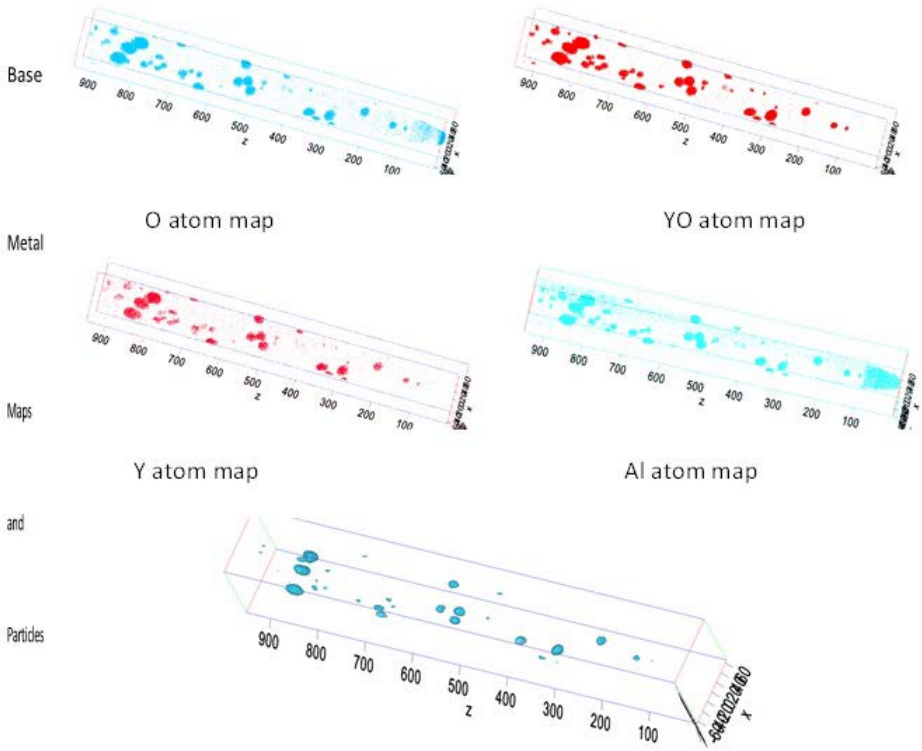

Figure 6. Atom maps and particles distribution in the base metal of ODS steel MA956.

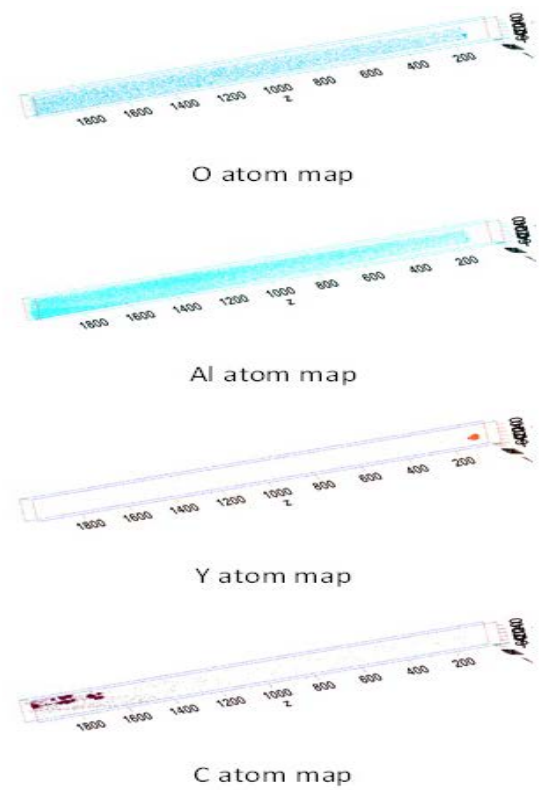

Figure 7. Atom maps showing the relatively uniform and dispersive distributions of major nano-oxide particle elements in the stir zone of ODS MA956.

Characterization of 14YWT

In ATP analysis of 14YWT, we used 5 at.% Ti iso-concentration surface to characterize nanoclusters since Ti is the major component in these nanoclusters. The result from base metal and stir zone is shown in Figure 7 and Figure 8 respectively. Stir zone shows much coarser nanocluster than base metal. Quantitative analysis on the nanoclusters shows the average radius of nanoclusters in base metal is 1.86 nm, comparing 2.83 nm in stir zone. There are two hypotheses on the coarsening of nanoclusters in stir zone. First, since friction stir welding is a process with high temperature and high deformation, there is a significant amount of vacancies generated in the stir zone during welding. Vacancies generated will accelerate the diffusion by several order of magnitude, which will lead to coarsening of nanoclusters in a very short period of time (several seconds). Second, nanoclusters may dissolve during stirring and re-precipitate during cooling. Vacancy-oxygen has a low bonding energy in steel. Excessive vacancies created by FSW increase the number of vacancy-oxygen and therefore increase the O solubility in steels. As a result, nanoclusters may dissolve during stirring as it was seen in MA956. As the temperature decrease during cooling, number of vacancies decrease. Nanoclusters may re-precipitate.

Synchrotron X-ray diffraction was also performed to characterize secondary phase in 14YWT. Figure 10 shows the comparison result between base metal and weld metal. Since the nanoclusters are very small, current study by X-ray diffraction did not reveal any O enriched nanoclusters. $M_{23}C_6$ carbides, on the other hand, were captured by X-ray diffraction in base metal. X-ray result shows fully dissolution of $M_{23}C_6$ carbides in the stir zone.

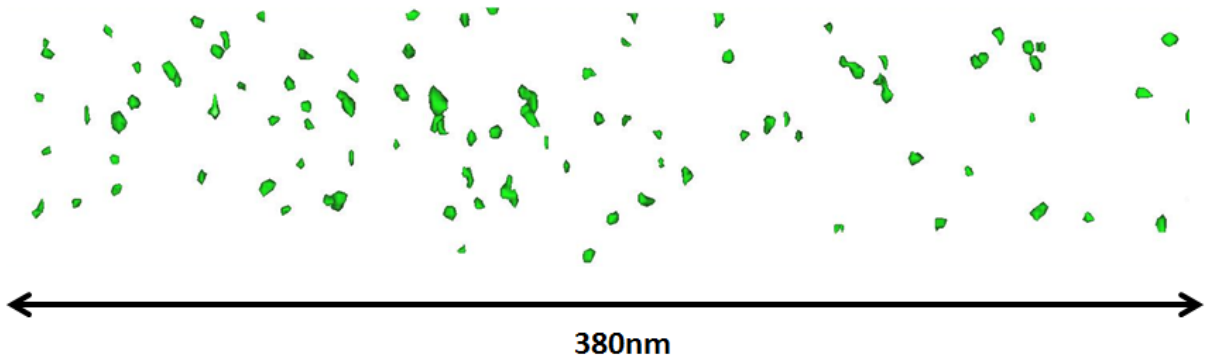

Figure 8. 5 at.% Ti iso-concentration surface of base metal showing nanoclusters enriched with Ti, Y and O

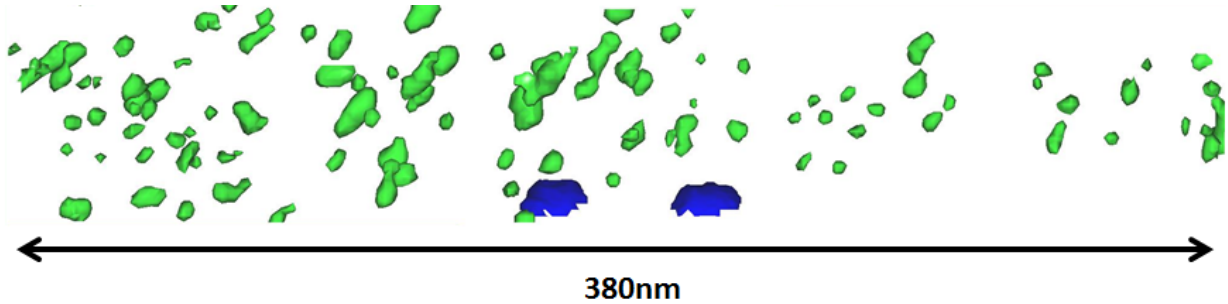

Figure 9. 5 at.% Ti iso-concentration surface of stir zone showing larger precipitates and inhomogeneous distribution

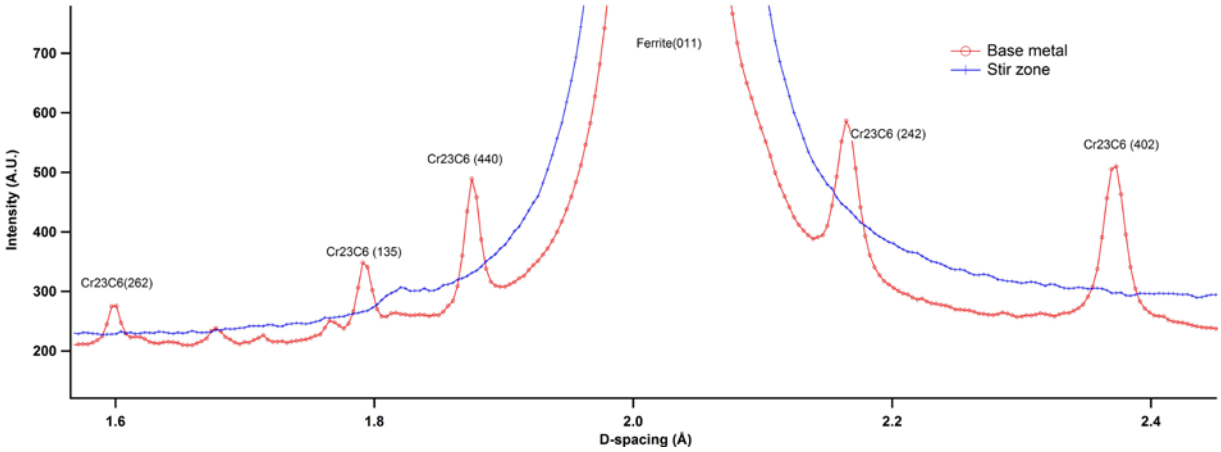

Figure 10. Synchrotron X-ray diffraction results from base metal and stir zone

Future Work

Consistent with the overall project plan, the following major activities are planned for the next 6 months:

- Additional FSW welds on RAFM steels will be produced with fine-tuned welding process conditions to further develop the basic understanding of process-microstructure-property relationship.
- Prepare for mechanical property testing including long-term creep testing, under the as-welded and different PWHT conditions.
- Additional in-situ DIC and IR measurements during FSW to obtain the temperature and stress information during FSW under different process conditions.
- Further APT characterization of nano particles/clusters in the friction stir welded ODS alloys (MA956 and 14YWT) including the effect of post weld heat treatment.
- Continue to investigate the bonding mechanisms between ODS alloys and RAFM steels through detailed microstructure characterization.
- Computation modeling to simulate the temperature and deformation history experience by the ODS and RAFM alloys.

Overall, the project is on schedule, without major technical issues.

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