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# Effect of friction stir welding and post-weld heat treatment on a nanostructured ferritic alloy☆

B. Mazumder [a,*], X. Yu [b], P.D. Edmondson [b], C.M. Parish [b], M.K. Miller [a], H.M. Meyer III [b], Z. Feng [b]

[a] *Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA*
[b] *Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA*

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ABSTRACT

Nanostructured ferritic alloys (NFAs) are new generation materials for use in high temperature energy systems, such as nuclear fission or fusion reactors. However, joining these materials is a concern, as their unique microstructure is destroyed by traditional liquid-state welding methods. The microstructural evolution of a friction stir welded 14YWT NFA was investigated by atom probe tomography, before and after a post-weld heat treatment (PWHT) at 1123K. The particle size, number density, elemental composition, and morphology of the titanium-yttrium-oxygen-enriched nanoclusters (NCs) in the stir and thermally-affected zones were studied and compared with the base metal. No statistical difference in the size of the NCs was observed in any of these conditions. After the PWHT, increases in the number density and the oxygen enrichment in the NCs were observed. Therefore, these new results provide additional supporting evidence that friction stir welding appears to be a viable joining technique for NFAs, as the microstructural parameters of the NCs are not strongly affected, in contrast to traditional welding techniques.

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## 1. Introduction

Nanostructured ferritic alloys (NFAs), such as 14YWT (Fe–14Cr–3W(wt.%) base ODS steel with additions of Ti and Y), are considered promising candidate materials for structural materials in fusion reactors and cladding materials of future advanced fast reactors, due to their high temperature creep properties and tolerance to high-dose radiation [1,2]. The excellent creep resistance and the radiation resistance of these alloys are mainly due to the high number density of ultrafine Ti–Y–O enriched nanoclusters (NCs) and ultrafine grain size (<500 nm) [3–5]. In particular, oxide dispersion strengthened (ODS) steels (and by extension NFAs), are strong candidate materials for fusion blanket designs [7–9]. Blankets will have complex piping and other fabrication requirements and will require a means to weld NFAs to NFAs and NFAs to other steels, especially considering the difficulty in making large monolithic sections from NFAs. This is difficult because NFAs are produced by a mechanical alloying (MA) process to force the Y and O from the $Y_2O_3$ powder into solid solution, in order to form ultrafine Ti–Y–O NCs upon subsequent thermal processing steps [6]. Due to this requirement to use the MA process to fabricate these alloys (rather than conventional casting) [10], it is not technically feasible to produce complex, or large-scale, components. Therefore, there is a requirement to develop joining techniques for similar and dissimilar materials for NFAs, e.g. 14YWT-14YWT; and 14YWT-F82H. Currently, the lack of any joining technique that maintains the properties of welded structure as good as base metal limits the application of these alloys. Conventional fusion (liquid-state) welding destroys the fine dispersion of oxide particles, causes grain coarsening, and modifies the dislocation structures in the alloy [11,12] and so is not applicable. Hence, solid-state methods must be used. Friction Stir Welding (FSW) has been considered to be a

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* Corresponding author. Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6139, USA.
*E-mail address:* mazumderb@ornl.gov (B. Mazumder).

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favorable technique to weld NFA alloys, while preserving the advantageous microstructure [12–14]. FSW uses a high-strength rotating tool inserted into the workpiece to make a joint without melting, which gives a strong weldment with minimum degradation of the microstructure and microscopic properties.

An important question that must be answered is how the microstructure and chemical composition of the ultrafine dispersoids is modified after the FSW process. A variety of studies have been carried out, including electron microscopy [9,12,14,15], to identify NC coarsening and their distribution throughout the weld zone [12,9,16]. During the FSW process in the stir zone, the original microstructure of the work piece is affected both by heat and plastic deformation, and a modified oxide-dispersed microstructure formed. Different zones, namely base metal (BM), stir zone (SZ), thermo-mechanically affected zone (TMAZ), and heat-affected zone (HAZ) are usually reported [17]. These modified microstructures from the different regions of welded sample need to be evaluated for their chemical and structural morphology in order to understand the related material properties, particularly how the NCs are modified and how their morphology/chemical compositions are altered. This information will help explain the overall macro-scale properties of the material, gauge the effective quality of the weld, and the suitability of the joint for nuclear applications.

Previous research has proven that FSW is a viable technique to join ODS alloys, including NFAs [18–21]. ODS alloys, such as MA956, showed significant coarsening of the oxides after FSW, and concomitant loss of strength in the SZ [19,20]. In NFAs 14YWT, small NCs were observed after the FSW, and showed significant variations in hardness across the FSW [21]. Atom Probe Tomography (APT) has been successfully used to provide detailed structural information, such as the particle size and distribution, number density, and the composition of these nanostructures from the different zones. Both coarser-structured ODS alloys [22] and finer-structured NFAs [21] have shown modification of the precipitates after welding and post-weld heat treatment (PWHT). The microstructure in the SZ and TMAZ shows fine ferrite grains with high densities of ultrafine Ti–Y–O-enriched precipitates [21,22]. However, due to heat and deformation induced by the FSW process, SZ and TMAZ also showed non-uniform distribution of NCs, which result in a reduction of strength in these regions. It should be noted that, as the studies above used different FSW process conditions, the difference in the observed stability of the nano-sized precipitates could also be attributed to the variation in temperature and deformation in these experiments, in addition to the difference in the types of the nanoprecipitates. As PWHT is a standard procedure for weld strength recovery, the current study examines the effect of PWHT on the microstructure of FSW 14YWT NFA.

## 2. Experimental procedure

The material examined in this investigation was a 14YWT NFA [23]. The 14YWT NFA was produced by a mechanical alloying process by ball milling pre-alloyed Fe–14Cr–3W–0.4Ti powder with 0.3 wt. % $Y_2O_3$ powder. The resultant powder was subsequently degassed in a sealed steel can at 673 K and extruded at 1123 K into a rod. The rod was then annealed at 1273 K for 1 h and fabricated into plate by rolling to 40% reduction in thickness, 2.86 mm, at 1123K [21]. The bulk composition of this material is shown in Table 1.

**Table 1**
Composition of 14YWT NFA. The balance is Fe.

| 14YWT | Cr | Y | W | Ti | O | C | Si | N |
|---|---|---|---|---|---|---|---|---|
| at. % | 13.93 | 0.14 | 0.16 | 0.24 | 0.39 | 0.24 | 0.16 | 0.15 |
| wt. % | 13.13 | 0.22 | 0.54 | 0.19 | 0.12 | 0.05 | 0.08 | 0.04 |

The friction stir welding was conducted in a MTS ISTRI friction stir machine with a polycrystalline cubic boron nitride (PCBN) pin, as shown schematically in Fig 1. The PCBN pin had a shoulder diameter of 16 mm with a threaded, nominally 6 mm diameter, pin. The length of the pin was 3 mm, i.e., slightly longer than the thickness of the 14YWT plates. A 2.86 mm thick plate of F82H steel with a nominal composition shown in Table 2 was used as a support substrate. The tool had a rotation speed of 300 rpm and traverse rate at 7.62 cm per min. The rotating pin tool traveled parallel to the extrusion and rolling directions of the 14YWT specimens, as shown in the previous work [21]. Part of the resulting FSW sample was then PWTH at 1123K for 2 min.

APT specimens were prepared from site-specific locations in the SZ, TMAZ and BM from both the as-FSW and PWHT samples with a FEI Nova 200 Focused Ion Beam ion miller. The specimens were analyzed in a CAMECA Instruments Inc. Local Electrode Atom Probe (LEAP 4000X HR) in pulsed-laser mode. The data was collected from specimens that were cooled to a temperature of 30 K under ultrahigh vacuum ($<10^{-11}$ mbar). To achieve controlled field evaporation, short-duration, ultraviolet (UV) laser pulses with laser energy of 100 pJ were focused on the apex region of the specimen. Note that the diameter of the laser beam was significantly larger than the apex region of the APT specimen, so not all this energy was introduced into the specimen. A pulse repetition frequency of 200 kHz and a detection rate of 0.005–0.01 atoms per pulse were also used. Data reconstruction and analysis were performed with the CAMECA Instruments Inc. IVAS (version 3.6.6). Atom probe tomography was performed on the three regions BM, SZ and TMAZ, before and after heat treatment (HT) for a structural comparison.

The volumes of the Ti–Y–O precipitates, estimated from the isosurface are converted to the effective radii using the equation, $r_{eff} = \sqrt[3]{3V/4\pi}$, assuming spherical geometry. The number densities were estimated from the number of fully-contained plus half the partially-contained precipitates within the volume, the number of ions in the assigned peaks of the mass spectra, the detection efficiency of the mass spectrometer (37%) and the volume of the standard 2 atom body-centered cubic unit cell of Fe [24].

X-ray photoelectron spectroscopy (XPS) was performed with a Thermo Scientific Model K-Alpha XPS instrument. The instrument utilizes monochromated Al $K_\alpha$ X-rays (1486.6 eV) focused to a 400 μm spot size for maximum signal and to obtain an average surface composition over the largest possible area. The instrument has a hemispherical electron energy analyzer equipped with a 128 multi-channel detector system. Base pressure in the analysis chamber is typically $2 \times 10^{-9}$ mbar or lower. Samples were mounted to the sample platen using metal clips and an area within the weld zone was analyzed on each sample. Survey spectra

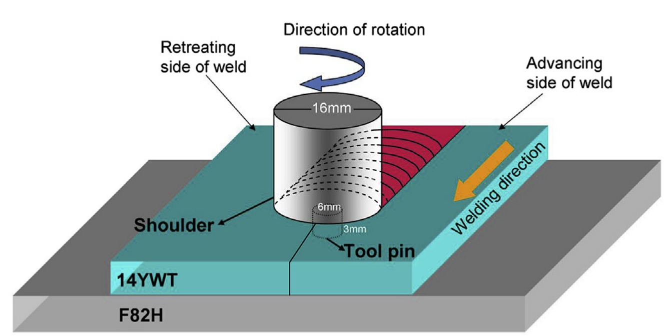


**Fig. 1.** Schematic illustration of the friction-stir welding process after [40]. A F82H base plate was used due to the limited thickness of the 14YWT plates.