Article

Solid-State Welding of the Nanostructured Ferritic Alloy 14YWT Using a Capacitive Discharge Resistance Welding Technique

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Abstract: Joining nanostructured ferritic alloys (NFAs) has proved challenging, as the nano-oxides that provide superior strength, creep resistance, and radiation tolerance at high temperatures tend to agglomerate, redistribute, and coarsen during conventional fusion welding. In this study, capacitive discharge resistance welding (CDRW)—a solid-state variant of resistance welding—was used to join end caps and thin-walled cladding tubes of the NFA 14YWT. The resulting solid-state joints were found to be hermetically sealed and were characterized across the weld region using electron microscopy (macroscopic, microscopic, and nanometer scales) and nanoindentation. Microstructural evolution near the weld line was limited to narrow (~50–200 µm) thermo-mechanically affected zones (TMAZs) and to a reduction in pre-existing component textures. Dispersoid populations (i.e., nano-oxides and larger oxide particles) appeared unchanged by all but the highest energy and power CDRW condition, with this extreme producing only minor nano-oxide coarsening (~2 nm → ~5 nm Ø). Despite a minimal microstructural change, the TMAZs were found to be ~10% softer than the surrounding base material. These findings are considered in terms of past solid-state welding (SSW) efforts—cladding applications and NFA-like materials in particular—and in terms of strengthening mechanisms in NFAs and the potential impacts of localized temperature–strain conditions during SSW.

Keywords: solid-state welding; capacitive discharge resistance welding; pressure resistance welding; oxide-dispersion-strengthened; nanostructured ferritic alloys; cladding

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1. Introduction

Clean and affordable next-generation nuclear power systems are essential ingredients to achieve energy security and meet targets for greenhouse gas reduction. Despite their promise, such systems expose materials to extremes of radiation, temperature, and corrosive or reactive environments that require novel advances in alloy design and processing [1,2]. The development of oxide-dispersion-strengthened (ODS) alloys in recent decades has provided a number of promising candidate materials, including MA956, PM 2000, and Kanthal APMT, with an improved tolerance to elevated temperatures and radiation damage [3–8]. Improvements in alloy chemistry and processing have since reduced dispersoid sizes from tens of nanometers to less than 5 nm, giving rise to a class of nanostructured ferritic alloys (NFAs) [9–13]. NFAs contain the pyrochlore $Y_2Ti_2O_7$ and orthorhombic Y_2TiO_5 particles that are formed using high-energy mechanical alloying, consolidation, and heat treatment of steel powders with small quantities of titanium and yttria (Y_2O_3). These dispersoids allow for NFAs to improve on the performance of other ODS materials by pinning grain boundaries and dislocations—for superior grain size stability and creep

resistance at high temperature [9,14,15]—and by providing recombination and trapping sites for radiation-induced point defects and helium—for reduced swelling, embrittlement, and chemical segregation with irradiation [16–21].

Unlike the secondary phase particles used to strengthen conventional wrought materials (e.g., carbides, γ'/γ'' in austenitic steels), Y-Ti-O nano-oxides do not dissolve into solution at working temperatures [22–24] and have been found to be extremely stable under both neutron and ion irradiations between 300 °C and 450 °C [25–34]. While this stability underpins the excellent performance of NFAs under extreme conditions, it creates a particularly severe challenge when joining NFA components using techniques that involve melting, such as traditional fusion welding (e.g., arc or laser welding) or transient liquid phase bonding. The Y-Ti-O nano-oxides are not soluble in the liquid phase of the NFA, instead tending to agglomerate together or redistribute to surfaces in the melt region [35–40]. Further, excess heat deposited during these techniques creates large thermal gradients in the heat-affected zones (HAZs) of each component that can promote the diffusion and coarsening of otherwise stable microstructures. While neither of these effects prevent the creation of solid joints, properties for the weld-affected material (e.g., melt region, HAZs) are comparable to the non-ODS material, defeating the advantages of NFAs.

Solid-state welding (SSW) techniques largely avoid these issues with NFAs due to the obvious lack of melting and lower peak temperatures, and thus tend to produce better welds. Several common and well-developed mechanical SSW methods have been proven as viable for joining ODS steels and NFAs, including friction welding (FRW) [41–43] and friction stir welding (FSW) [44–49]. However, some coarsening, strain-induced agglomeration, and inhomogeneous redistribution of nano-oxides have been observed in the stir zones and thermo-mechanically affected zones (TMAZs) produced during these techniques [41,47,50,51]. While this degradation is still considerably less than for fusion processes, FRW and FSW are made less attractive by their high tooling costs, restriction to simple weld geometries, inability to produce hermetically sealed joints, and their still considerable periods at elevated temperature (several seconds) for joint material. Solid-state pressure resistance welding (PRW) techniques offer strong alternatives to this mechanical mixing approach through innovations on electrical resistance welding—itself a 19th century adaptation. Such methods use Joule heating to soften the material, while the simultaneous compression of the components against each other forms the joint. PRW is often improved through the use of a thinned contact point, known as a “projection”, between the components being joined. The reduced contact area of the projection focuses Joule heating at the point of contact, leading the projection to soften before surrounding material and to collapse during compression. The softened material from the collapsing projection flows outwards across the weld surfaces, filling the joint and displacing surface oxides and debris without melting [52,53]. Capacitive discharge resistance welding (CDRW) shares many aspects of design with these PRW variants and is differentiated primarily by the power source and use of a pneumatic, fast follow-up system. Electrical currents are supplied continuously for PRW, for both pre-heating and joining components, while a discharging capacitor network provides a discrete pulse of current for CDRW. These methods offer advantages over other SSW processes in terms of time per joint (<100 ms), extreme localization of weld effects, and flexibility in weld geometry.

The successful joining of thin-walled cladding tubes to end caps has been reported by Zirker et al. [54] and Bottcher et al. [55] (NFA MA957 and a proprietary alloy PNC-ODS), Seki et al. [56] (9Cr-ODS), Gan et al. [57] (Kanthal-D FeCrAl), Jerred et al. [58] (NFA MA957 to HT9 steel), and Doyen et al. [59] (9Cr and 14Cr NFAs) using PRW without projections. These findings are particularly significant given the push in recent years to develop cladding materials for use in advanced fast reactors and for loss-of-coolant-accident conditions in commercial light water reactors. In this work, we explored the use of CDRW to join cladding tubes and end caps made from the NFA 14YWT. The rapid release of stored energy from a capacitor bank (i.e., high current, short duration pulses) was expected to allow for material flow and SSW with a significantly lower heat input—and thus, less evolution of the fine

grain structure ($<1\ \mu\text{m}\ \varnothing$) and nano-oxide dispersion ($\text{Y}_2\text{Ti}_2\text{O}_7$, $<10\ \text{nm}\ \varnothing$, $>10^{23}\ \text{m}^{-3}$) of 14YWT [27,60–62]. Although some experimental conditions produced welds that did not extend the full length of the joint or that expelled material into or out from the final capped tube, all welded samples were leak-tested to ensure a hermetic seal and were subjected to a qualitative petal test to guarantee the retention of strength and ductility at the weld. The specifics of the welding processing are addressed in a separate publication [63]. The weld performance was thus evaluated on the broad area of each joint that bonded fully and properly, with a focus on microstructural evolution (e.g., recrystallization, coarsening) and dispersoid stability.

2. Materials and Methods

Components for this study were produced using tubes and caps from the best practices heat of 14YWT developed by the Fuel Cycle Research and Development (FCRD) program, FCRD-NFA1 [64], the composition of which is shown in Table 1. Powder was produced by ATI Metals in Pittsburgh, PA using gas atomization and processed by Zoz, GmbH in Wenden, Germany using high intensity attritor milling for 40 h [65]. The base material rods were produced at Oak Ridge National Laboratory using hot extrusion at $850\ ^\circ\text{C}$ [66]. Cylindrical caps ($10\ \text{mm}\ \text{OD} \times 4\ \text{mm}$) were cut directly from the extruded rod using electrical discharge machining (EDM) and chamfered (45°) on one edge using a lathe. Tubing ($10\ \text{mm}\ \text{OD}$, $0.5\ \text{mm}$ thick) was produced by drilling out the center of the extruded rod and pilger processing the resulting tube at the French Alternative Energies and Atomic Energy Commission (CEA), Saclay. Four 40% reductions at room temperature were used to reach the final tube dimension, with intermediate annealing at $1200\ ^\circ\text{C}$. Individual tubes were then cut using EDM. Redeposited layers from EDM were removed from all electrical contact surfaces of the components (e.g., end surfaces of tubes and caps) using 1200 FEPA grit SiC paper. Despite producing caps and tubes from the same base material, grain size in the pilger processed tubing was significantly larger and more varied than in the cap material ($5\text{--}50$ vs. $\sim 0.5\ \mu\text{m}\ \varnothing$, smallest dimension). This difference is attributed to intermediate annealing during the pilger process. Processing heterogeneities were also observed for the cap material in the form of $\sim 100\ \mu\text{m}$ wide regions of enlarged grains ($10\text{--}50\ \mu\text{m}\ \varnothing$). Such veins lay along the extrusion direction and did not appear to affect the CDRW process or results.

Table 1. Composition of the NFA 14YWT FCRD-NFA1 [64].

Element	Fe	Cr	Y	W	Ti	O	C	Mn	Al	Cu	Ni
Concentration (wt%)	Bal	13.6	0.25	3.12	0.40	0.097	0.008	0.020	0.012	0.037	0.041

Caps and tubes were joined by Edison Welding Institute (EWI) in Columbus, Ohio. A detailed discussion of the CDRW welding system and process parameters is provided in the companion text [63], whereas the process is shown schematically in Figure 1a. Sample parts were mounted such that the inner edge of the tube wall was in contact with the chamfered outer edge of the cap, creating a self-centering and extremely narrow electrical contact for resistive heating. A precision triggering system was then used to simultaneously: (1) apply a compressive force ($3\text{--}4\ \text{kN}$) from a pneumatic load head equipped with pre-compressed Belleville washers for fast follow-up (i.e., uniform force over very brief times); and (2) deliver a high current ($15\text{--}34\ \text{kA}$), short rise time ($2\text{--}6\ \text{ms}$) electrical pulse from a transformer–capacitor power circuit similar to [52]. (Here, rise time refers to the time between pulse discharge and peak current.) An eight-tap transformer capable of winding ratios of 75:1 to 1625:1 was used to control the output pulse shape from the capacitor bank ($1.28\ \text{mF}$, up to $2.1\ \text{kV}$). This simultaneous compression and resistive heating was designed to produce material flow and solid-state bonding where the chamfered cap surface and inner tube edge met. Current waveform and displacement of the cap in the compression direction (CD)—the tube is fixed in place—were recorded for each sample,

as shown in Figure 1b. Weld conditions were described in terms of force, peak current, rise time, energy (i.e., total energy of the electrical pulse), and power (i.e., average energy rate of the electrical pulse). As peak current, rise time, energy, and power are all aspects of the electrical pulse shape, only two can be controlled independently (e.g., the rise time dictates the peak current and power for a given energy, and vice versa). Welded samples were screened first with visual inspection (e.g., for failure to bond, for obvious melting) and then using helium leak tests with a Veeco MS 40 system. Solid-state joints that demonstrated leak rates below 10^{-10} cc/s (air equivalent) were designated as hermetically sealed. Samples produced at the conditions listed in Table 2 consistently met both criteria and were selected for characterization and testing in this study. As no significant differences in microstructural evolution or mechanical behavior were observed between these weld conditions, the results in the following section are presented to encompass all conditions. Comparisons of Weld Conditions 1–3 are provided where appropriate to emphasize key findings (e.g., nano-oxide stability).

Figure 1. (a) A solid-state joint is formed from simultaneous pressure and resistive heating at a narrow contact point, shown in cross-section. (b) Representative pulse current and cap displacement measured during CDRW. Note that cap movement is delayed versus current, reflecting the time for material to heat and soften, and finishes before the pulse completely dissipates, indicating that the remaining current is insufficient to heat the broadened contact and sustain flow.

Table 2. Weld conditions for CDRW of 14YWT.

Weld Condition	Force (kN)	Peak Current (kA)	Rise Time (ms)	Energy (J)	Power (kW)
1	4.0	25	2.2	360	82
2	3.1	16	5.6	580	51
3	3.1	32	2.2	580	130

Welded samples were sectioned using EDM. Cross-section samples—similar to the flag-like shapes in Figure 1a—were polished with SiC papers to 2000 FEPA grit and finished with $0.04\ \mu\text{m}$ colloidal silica. The polished surfaces were etched for 15 s with modified aqua regia—a 3:1:1 mixture of hydrochloric, nitric, and lactic acids—to remove mechanical polishing damage and to preferentially etch grain boundaries for enhanced optical microscopy contrast. (The addition of lactic acid to aqua regia slows both the aging of the solution and the rate of attack, similar to glyceresia, and makes the modified solution a more consistent, user-friendly etchant.) For consistency, samples in this study will be described according to three directions: CD, the radial direction (RD), and a transverse direction (TD) that is orthogonal to the others.

Examination of the weld microstructure was performed on a coarse scale ($10\text{'s}\ \mu\text{m}$ –1 mm) using scanning electron microscopy (SEM) and electron backscatter diffraction

(EBSD) in a Thermo Scientific Apreo SEM with an EDAX VelocityTM detector and TSL software. Analysis of the weld line (the interface between joined cap and tube) and other fine features (<10 μm scale) was performed using transmission Kikuchi diffraction (TKD) on the same Apreo SEM. Dispersed oxides were imaged using transmission electron microscopy (TEM) and scanning TEM (STEM) in Thermo Scientific Tecnai F30 and Titan microscopes, both operating at 300 keV. Electron transparent foils for TKD, TEM, and STEM were produced using focused ion beam (FIB) milling in a Thermo Scientific Helios 600 FIB. Milling was performed at 30 kV with decreasing probe currents to a foil thickness of ~ 250 nm, followed by an intermediate thinning at 16 kV and a final polish at 2 kV. This last step was particularly important to reduce preparation artifacts on the sample surface. Foils were produced especially wide (~ 30 μm) to better understand how proximity to the weld line may effect microstructure. Large ODS dispersoids (>10 nm $\varnothing$) were imaged using Z-contrast in high-angle, annular dark-field (HAADF) STEM, whereas finer dispersoids were imaged using mass-thickness contrast and their characteristic shape under near zone axis, bright-field TEM. Energy dispersive X-ray spectroscopy (EDS) was used to examine the chemical make-up of larger dispersoids.

Nanoindentation was performed at the Center for Integrated Nanotechnologies (CINT) at Los Alamos National Laboratory [67], using a Keysight G200 Nanoindenter. A diamond, pyramidal (Berkovich) tip was used for indentation to a final displacement of 400 nm and at a constant strain rate (loading rate divided by the load) of 0.05 s^{-1} . Continuous stiffness measurements (CSM) were performed at a frequency of 45 Hz and 2 nm displacement amplitude. Hardness and modulus measurements were determined using the Oliver-Pharr method [68]. Hardness was profiled across the weld lines, parallel to CD. Each profile consisted of 40 indents spaced 10 μm apart, with more than 100 indents per sample. Positioning was controlled by centering the end of the weld line in the optical scope, moving the proper distance to the desired starting position for a row of indentations, and then proceeding to indent the sample up to and across the weld. While movement of the indenter stage may introduce some error in indent placement over large distances, this method was expected to be accurate enough to observe variations in hardness between weld zones and across the joint.

3. Results

3.1. Macro Characteristics

While the companion text [63] examines the macro-scale appearance and quality of these and similar welds in great detail, some points will be repeated here for context. In Figure 2, a low magnification photograph and EBSD maps of the samples showcase common features of the CDRW joints. No fusion zones were observed for the samples, as expected for a SSW technique, and alterations to the components were confined to ~ 50 – 200 μm thick TMAZs, highlighted using black dashed lines in Figure 2a. These TMAZs are differentiated from the unaffected materials of the cap and tube by the warping of pre-existing textures, with grains appearing to bend to lie parallel to the weld line, as shown in Figure 2b, and are thinner in the caps than in the tubes. The recrystallization and coarsening that characterize typical HAZs and TMAZs were not observed along the bulk of the weld line or in the adjacent TMAZs (i.e., the region roughly bounded by black dashed lines).

Recrystallization and coarsening were only observed to any significant extent in the expulsion regions at the inner and outer edges of the weld, as shown in Figure 2c–e. These defects did not compromise the quality of the welds (e.g., hermetic seal)—being excess material—and are only significant beyond the scope of this work (e.g., for CDRW process optimization). A brief examination is useful for later comparisons to the weld line and weld-adjacent microstructures that are of interest here. At the inner edges of the welds, as shown in Figure 2c, a new intermediate size, equiaxed, and texture-free grain structure is observed and attributed to recrystallization—possibly continuous dynamic recrystallization—during joining. Grains are frequently seen bending back on themselves (highlight 1) or breaking

up into roughly equiaxed sub-grains (highlight 2). At the outer edges of the welds, the appearance of coarsened material can range from enlarged or smeared grains coating a gap in the outer sample surface, as marked with arrows in Figure 2d, to beads or whiskers of expelled material, as shown in Figure 2e. While these presentations differ greatly, they are all attributed to slower cooling at the surfaces of the sample. The dispersed oxides from these expulsion regions are especially significant for comparison to those along the main weld lines, as they offer a look at agglomeration and coarsening effects in the material where heat input was not effectively controlled.

Figure 2. (a) Light optical microscopy photograph of a 14YWT tube and cap joined using CDRW with Weld Condition 3. Black dashed lines highlight the rough extent of TMAZs, whereas red dashed boxes highlight the sites of EBSD scans shown in (b,c,e). (b) Grain orientation map (relative to TD) showing component textures warping toward the weld line. Highlights 1 and 2 mark especially clear bending of grain groups in the cap TMAZ, whereas highlight 3 indicates an example of enlarged cap grains caused by the heterogeneity of hot extrusion—rather than CDRW with dashed lines added to highlight TMAZs. (c–e) Grain orientation maps (relative to TD) from grain recrystallization and coarsening defects in the expulsion regions of CDRW joints. Here, (d) comes from a joint produced at Weld Condition 1.

3.2. Weld Lines and Texture

The limited microstructural evolution revealed using macro-scale characterization was further investigated and confirmed using higher resolution EBSD and TKD scans. Higher resolution EBSD scans, such as that shown in Figure 3a, reveal cap and tube microstructures (i.e., grain sizes, textures) meeting at a mostly flat plane (dashed line). While grains at and near the weld line still do not appear coarsened, small groups of recrystallized grains are

occasionally seen clustered against the cap–tube interface (highlight 1). These clusters could be attributed to recrystallization, similar to the weld defect in Figure 2c, and suggesting the existence of hot spots in the interior of the joint during CDRW, or they could be surviving heterogeneities from the base materials. In either case, such clusters account for a very small fraction of the weld line and are not expected to impact the overall joint performance. Concerns with preparation artifacts in these scans (i.e., that poor patterns might falsely reduce grain sizes or mask structures in the cap material) were assuaged through TKD scans of material taken across the weld line, in the cap near the weld line (~10 μm away), and in the tube near the weld line, as shown in Figure 3b–d, respectively. The sharpness of the interface between components is especially apparent here (dashed line), given the clear differences in grain size between the cap and tube.

Figure 3. (a) EBSD grain orientation map (relative to TD) showing the lack of significant grain recrystallization or coarsening at the weld line. Highlight 1 marks a cluster of recrystallized grains along the weld line, whereas highlight 2 indicates another example of enlarged cap grains caused by the heterogeneity of hot extrusion. (b–d) TKD grain orientation maps showing material across the weld line, in the cap near the weld (~10 μm away), and in the tube near the weld, respectively. Here, all maps were collected from joints produced using Weld Condition 1 and colored according to the crystal orientation parallel to the indicated viewing direction for each map. Note the difference in sample orientation (i.e., frame of reference) between (b,c) and the accompanying change in appearance for the cap grains.

Orientation information from EBSD and TKD scans revealed changes in texture between the base materials and the TMAZs of the joint. Pole figures generated from the unaffected base materials (i.e., far from the joint), as shown in Figure 4a,b, display a shear deformation texture [69] for the pilgered tube and an α -fiber texture for the hot extruded cap. In both cases, there was a strong alignment of the $\langle 110 \rangle$ crystallographic direction parallel to CD. These textures are absent from pole figures generated from the tube and cap materials adjacent to the weld line, shown in Figure 4c,d. The apparent lack of texture in the near-weld tube could be due to limited sampling, but a sufficient number of cap grains were scanned to ensure the capture of a dominant texture—if present. Given the bending of grains in the TMAZs to lie parallel to the weld line, the texture was also considered relative to the plane of the weld line, marked using dashed lines in Figure 4c,d. While

such a texture might exist in the near-weld tube, it is significantly weaker than in the original components.

Figure 4. (a,b) Pole figures showing $\langle 110 \rangle$ alignment with the sample directions (CD, RD, TD) in the unaltered base tube and cap materials, respectively. (c,d) Comparable pole figures generated from material in the TMAZ of each material. Dashed white lines have been added to highlight the plane of the weld line. Texture intensity, I , has been plotted using a logarithmic color scale to highlight the extreme shift between base and TMAZ materials. Here, pole figures are taken from a joint produced using Weld Condition 3.

3.3. Dispersoids

TEM- and STEM-based characterization revealed minimal changes to dispersed oxide distributions (i.e., size, number density), consistent with the limited microstructural evolution observed at macro- and micro-scales. In Figure 5a, HAADF STEM images highlight the lack of variation in larger dispersoids (i.e., ~ 10 – 50 nm $\varnothing$ dark particles on the light background) across a representative joint. While these larger particles are not the critical nano-oxides discussed previously, they demonstrate a lack of obvious agglomeration or redistribution during CDRW. Size histograms for these larger particles are shown in Figure 5b. Although particles were occasionally recorded with diameters up to 50 nm, these distributions were consistently smaller than reported for other work with 14YWT (with sizes $\gtrsim 100$ nm) [70]. The slight size variations seen in Figure 5 are consistent with variations in the base materials, suggesting that it is not a CDRW-driven effect. Higher magnification imaging of these areas using bright-field TEM, as shown in Figure 6a, revealed nano-oxides as small as 2 nm $\varnothing$ within ~ 10 μ m of the weld lines. The squared appearance of the nano-oxides, highlighted in Figure 6c, suggests the presence of low-energy, semi-coherent interfaces with the NFA matrix—likely cube-on-cube ($\{100\}\text{Fe} \parallel \{100\}\text{Y} - \text{Ti} - \text{O}$, $100\text{Fe} \parallel 110\text{Y} - \text{Ti} - \text{O}$) or cube-on-edge ($\{100\}\text{Fe} \parallel \{110\}\text{Y} - \text{Ti} - \text{O}$, $100\text{Fe} \parallel 100\text{Y} - \text{Ti} - \text{O}$) orientation relationships described in previous reports [60,71]. These interfaces (i.e., the sides of the “squares”) become evident when imaging near $\langle 100 \rangle$ or $\langle 110 \rangle$ zone-axes.

A particular effort was made to examine dispersed oxide distributions from CDRW joints produced at varied weld energies and powers, given the importance of heat input to SSW performance [52,63,72]. Comparisons of Weld Conditions 1 (“cold”) and 3 (“hot”) are shown in Figure 5a vs. Figure 5c for larger dispersoids and in Figure 6a vs. Figure 6b

for nano-oxides. While the larger dispersoids appear qualitatively similar in these joints (i.e., size range, uniform distribution), nano-oxides appear larger in the Weld Condition 3 material (~ 5 nm vs. ~ 2 nm $\varnothing$), as presented in the nano-oxide size distributions in Figure 6d. This change in nano-oxides, but not larger particles, could be attributed to the very short duration of the CDRW process ($\lesssim 20$ ms), with sufficiently high local temperatures for Y and Ti diffusion but an insufficient time for long-range transport. However, it must be noted that the hot sample foil imaged here is also thicker ($\sim 2\times$) than the Weld Condition 1 material. Due to the fact that the visibility of nano-oxides depends on sample thickness (i.e., less fraction of the thickness $\rightarrow$ less contrast), the observed difference in size distribution may be artificial and the smallest nano-oxides (i.e., < 2 nm $\varnothing$) may be undercounted in general.

Figure 5. (a) HAADF STEM images of dispersoids (dark particles on the light background) across a CDRW joint formed using Weld Condition 1 (“cold”). Note the lack of agglomeration or redistribution (i.e., particles remain smooth and isolated). (b) Histograms of dispersoid size (diameter) for the regions shown in (a), based on 1380 particles. While particles with diameters greater than 20 nm were occasionally recorded, these are omitted from the plot for clarity. (c) Representative HAADF STEM image of dispersoids near (~ 5 μm) a CDRW joint formed using Weld Condition 3 (“hot”). Note the qualitatively similar size and uniform distribution of dispersoids at the nominally hotter Weld Condition 3.

Figure 6. (a,b) Bright-field TEM images of cap material within 10 μm of CDRW joints formed using Weld Conditions 1 (“cold”) and 3 (“hot”), respectively. Nano-oxides appear as grey, squared features—highlighted for clarity in (c)—due to imaging near $\langle 100 \rangle$ zone-axes. (d) Histogram of dispersoid size (diameter) for nano-oxides in these regions, based on 1223 particles. Note that smaller particles ($\lesssim 2$ nm $\varnothing$) may be undercounted due to imaging limitations, especially in thicker foils from the Weld Condition 3 joint.

While future process optimization is expected to reduce the expulsion regions discussed previously (Figure 2c–e), dispersoids from the outer expulsion regions—material where heat input was not effectively controlled—were studied for a comparison to less altered regions along the weld lines. Elemental maps showing a combined Y and Ti signal are presented in Figure 7 for samples taken from two such regions, highlighting representative dispersoid populations in each (note the difference in scale). As described previously, larger dispersoids in the weld line material were uniformly distributed and roughly spherical. In contrast, dispersoids in the expelled material were clearly larger, with the largest particles exceeding 100 nm $\varnothing$ and having very irregular (i.e., non-spherical) shapes. This difference in appearance is attributed to the agglomeration of pre-existing dispersoids in the latter material during CDRW, and suggests that such regions were at least partially molten. Further, the expected co-location of Y and Ti was observed for dispersoids

from both regions, regardless of particle size, making the misidentification of weld debris (e.g., titanium carbides or nitrides) unlikely.

Figure 7. Maps of the combined Y and Ti EDS signal showing the larger dispersoids found in material near the weld line (a) and from an outer expulsion region (b) of a joint produced using Weld Condition 1 (contrast has been inverted for improved visibility, with strong signal points appearing dark and weaker points appearing light). Note the difference in scales and the enlargement and roughening of particles shown in (b). Arrows have been added to highlight similar sized dispersoids in each scan.

3.4. Nanohardness

Measurements of nanohardness were compiled from multiple cross-section samples of CDRW joints and are plotted in Figure 8 with respect to the weld line. As expected, base material nanohardness was higher in the smaller-grained cap material than in the tube (~6.0 vs. ~5.5 GPa, respectively). More interesting, however, is the ~10% softening revealed by measurements in the cap and tube TMAZs (orange and blue shading, respectively). This effect was seen across the CDRW samples, regardless of the weld condition. Given the minimal grain evolution (i.e., lack of recrystallization, coarsening) noted previously and the apparent insensitivity to dispersoid changes (i.e., the slight nano-oxide coarsening at Weld Condition 3), the observed softening was initially attributed to a loss of dislocation density in the TMAZs during CDRW. A direct confirmation of this point was attempted using bright-field TEM imaging, as shown in Figure 9. While both as-pilgered (i.e., base material, never welded) and TMAZ (within 20 μm of the joint) tube materials contain visible dislocation forests, the apparent dislocation density is possibly lower in the post-CDRW material ($\sim 1.9 \times 10^{14} \text{ m}^{-2}$ vs. $\sim 2.2 \times 10^{14} \text{ m}^{-2}$). However, these observations should be treated qualitatively, as significant variations in the dislocation density were observed between the grains of each sample.

Figure 8. Nanoindentation measurements with distance from the weld lines of CDRW joints, based on 720 measurements across six joints produced using Weld Conditions 1–3. Note the expected difference in hardness between cap and tube materials (~6.0 vs. ~5.5 GPa) and the noticeable softening of material in the cap and tube TMAZs (shaded).

Figure 9. Bright-field TEM images of the 14YWT tubing used in this study, showing a possibly greater dislocation density in as-pilgered base material (a) than in post-CDRW material within 20 μm of a joint (b) formed using Weld Condition 3. The $g = \langle 011 \rangle$ diffraction vector was excited for imaging.

4. Discussion

A comparison to past SSW studies demonstrates that the TMAZs of these CDRW joints—although not the thinnest weld zones reported for 14YWT—are notably thin. The largest 14YWT weld zones, ~ 1 mm wide, were reported by Hoelzer et al. [73] and Mazumder et al. [51] after FSW, whereas Radhakrishnan et al. [74] observed HAZs as thin as a few microns after spark plasma (SP) joining thin plates. Among PRW studies of cladding, the appearance of weld zones is mixed. HAZs were not observed in the work of either Seki et al. on 9Cr-ODS steel [56] or of Gan et al. on Kanthal-D FeCrAl [57], whereas mixed MA957-HT9 joints produced by Jerred et al. [58] contained both HAZs and TMAZs (~ 500 μm wide total) on the HT9 (non-ODS) side of the weld and only TMAZs (< 100 μm wide) on the MA957 (ODS) side. These findings appear scattershot at first glance, but become clear when deformation and heat input are considered together. The TMAZs seen for FSW are absent for SP because the latter uses minimal compressive force; the mixed PRW samples show HAZs not found in the single material joints because the penetration of heat and deformation (relative to one-another) is distinct for each material. The behavior of 14YWT joints studied here (e.g., lack of HAZs, differences in TMAZs between caps and tubes) can thus be understood as a necessary—and positive—consequence of the lower heat input of our CDRW technique.

Considering this lower heat input raises interesting comparisons to previous work by Corpacci et al. [72] on the weldability of PM2000 ODS cladding using PRW. Corpacci described several regimes of energy dissipation (i.e., heat input), observing increases in the un-bonded weld line length at a lower dissipation and increases in dispersoid redistribution at a higher dissipation. While incomplete bonding is not a directly comparable metric—as the weld force and joint design strongly affect it, the occasional observation of un-bonded length at the edges of these CDRW joints and the absence of significant microstructural or dispersoid modification suggest that these conditions lie in a similar low energy dissipation regime. The conditions studied here cannot provide any additional insight into where the high dissipation regime, with excessive recrystallization and coarsening, may begin. As before, readers are referred to the companion text [63] for a more complete consideration of the role welding parameters played in forming these CDRW joints.

The most direct comparison available for these CDRW samples comes from a study by Doyen et al. [59] on the PRW of 9Cr and 14Cr NFA claddings. Doyen’s 14Cr caps and tubes were cut from a small-grained, α -fiber textured (extruded) base material—similar to the 14YWT caps in this study—and formed joints without HAZs—in line with the 14YWT CDRW joints here. The reported TMAZs in their joints were slightly thicker than in this

study (~250 vs. ~200 μm) and revealed a gradual transformation from the α -fiber texture to a shear texture aligned with the radial sample direction (i.e., a differently aligned version of our base tube texture). Doyen attributed this change in texture and the accompanying disappearance of elongated grains to dynamic recrystallization during PRW, in contrast to the grain bending—but not recrystallization—seen here for CDRW. Unfortunately, nano-oxides in these NFAs were not inspected using TEM but were characterized using small-angle X-ray scattering (SAXS). These SAXS measurements revealed an increase in the diameter of nano-oxides in the TMAZs from ~2 nm to ~7 nm, similar to the enlarged nano-oxides observed in CDRW samples produced at Weld Condition 3 (~5 nm post-CDRW vs. ~2 nm initially). This coarsening and Doyen's observation of dynamic recrystallization suggest a higher heat input for their PRW process than for all but the highest energy and power CDRW conditions studied here.

The dispersoid stability observed for these CDRW samples was consistent with and sometimes better than prior SSW reports. The previously discussed study by Hoelzer et al. [73] found no coarsening or agglomeration of dispersoids in the FSW weld zones, whereas the similar work by Mazumder et al. [51] revealed the formation of larger, elongated dispersoids (i.e., filaments) in the stir zone. Mazumder also characterized the dispersoid distributions after post-FSW heat treatment, revealing a significant shift in the nano-cluster population versus the base material (~0.5x $\rightarrow$ 4x); distributions in the base and TMAZ materials were more stable. This ~10x increase was attributed to either the fresh nucleation of dispersoids or to the widespread coarsening of previously unobservable nano-clusters, but it could be an artifact from the use of atom probe tomography to count relatively small (<2 nm $\varnothing$) dispersoids via concentration isosurfaces. Although the potential changes to 14YWT during service (e.g., annealing, irradiation) concerned Mazumder, the differences from other FSW work suggest a unique deformation state that is unlikely to affect other SSW techniques.

Changes in mechanical behavior across SSW joints have been frequently reported and are typically attributed to phase refinement (e.g., austenitization in steels with $\lesssim 13$ wt% Cr followed by martensitic transformation), grain coarsening, dispersoid agglomeration, dislocation density loss during recrystallization, and porosity due to suboptimal welding [44,45,56,58,59,73]. The ~10% softening observed for these CDRW joints is relatively mild compared to the 7–35% softening in such reports, and the lowest hardness measurements here (~5.0 GPa) are still considerable compared to those of other advanced cladding materials, such as HT-9 (~2.6 GPa) or MA957 (~4.0–4.8 GPa). The softening observed here for cap and tube TMAZs is broadly consistent with such reports and is most directly comparable to the previously mentioned studies by Doyen [59] and Jerred [58]. While both studies reported martensite-induced hardening for their ferritic–martensitic steels (9Cr and 12Cr, respectively), results for the 14Cr materials differed, with a near constant hardness observed by Doyen and a ~150 μm wide region of softening measured by Jerred. This discrepancy could be artificial, given that both used microhardness testing at a spacing of 50 μm .

Assuming that reports by Doyen and Jerred are correct leads to some interesting comparisons with this study. The approximately equivalent regions of softened MA957 and TMAZ reported by Jerred mirror the softening of 14YWT in this study and suggest that grain bending during CDRW is similar in effect to recrystallization. Comparisons between the 14YWT caps studied here and the extruded components used by Doyen are also particularly interesting, as the nano-oxide coarsening reported by Doyen would be expected to produce softening in their joint TMAZs, but did not. This finding could be explained by the recrystallization-induced grain refinement (4.6 μm to 1.5 μm $\varnothing$) in those same TMAZs, with an increase in grain boundary strengthening compensating for a reduction in the dispersoid contribution. Taken together, the recrystallization effects reported by Jerred (apparently detrimental) and Doyen (potentially restorative) suggest a varied and not well understood role in the SSW of 14YWT and other advanced alloys.

Further study of this point may be warranted in order to determine if recrystallization could be controlled to produce improved joints with these cladding materials.

5. Conclusions

In this study, CDRW was used to form hermetically sealed, solid-state joints between 14YWT NFA caps and tubes, simulating the closure of advanced nuclear fuel cladding. Key findings included:

- Microstructural evolution in the welded components was limited to ~200 µm thick TMAZs; lacked obvious recrystallization, coarsening, or agglomeration of grains or dispersoids reported for other joining methods (conventional and SSW); and suggests a relatively low energy dissipation; and
- Softening was measured during the nanoindentation of the TMAZs despite this apparent lack of microstructural change, was minor compared to past joining studies, and left sufficient hardness to be comparable with or stronger than other advanced cladding materials.

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Abbreviations

CD	Compression direction
CDRW	Capacitive discharge resistance welding
CEA	French Alternative Energies and Atomic Energy Commission
EBS	Electron backscatter diffraction
EDM	Electrical discharge machining
EDS	Energy dispersive X-ray spectroscopy
EWI	Edison Welding Institute
FCRD	Fuel Cycle Research and Development
FIB	Focused ion beam
FRW	Friction welding
FSW	Friction stir welding
HAADF	High-angle annular dark field
HAZ	Heat-affected zone
NFA	Nanostructured ferritic alloy
ODS	Oxide-dispersion-strengthened

PRW	Pressure resistance welding
RD	Radial direction
SAXS	Small-angle X-ray scattering
SEM	Scanning electron microscopy
SP	Spark plasma
SSW	Solid-state welding
STEM	Scanning transmission electron microscopy
TD	Transverse direction
TEM	Transmission electron microscopy
TKD	Transmission Kikuchi diffraction
TMAZ	Thermo-mechanically affected zone

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